A STUDY OF THE EFFECTS OF SCOURING AND MOISTURE CONDITIONING ON THE RATE AND DEGREE OF PARTIAL ACETYLATION OF SEVERAL VARIETIES OF COTTON FIBERS

A THESIS

Presented to

the Faculty of the Graduate Division

by

Sherman Wesley Blandin, Jr.

In Partial Fulfillment

of the Requirements for the Degree

Master of Science in Textile Engineering

Georgia Institute of Technology

June 1953

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I wish also to acknowledge the assistance of the Southern Regional Research Laboratory of the U. S. Department of Agriculture for furnishing the cottons used, and of the Chemistry Department of the Research Division of the West Point Manufacturing Company, West Point, Georgia for furnishing some of the data presented.

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A STUDY OF THE EFFECTS OF SCOURING AND MOISTURE CONDITIONING ON THE RATE AND DEGREE OF PARTIAL ACETYLATION OF SEVERAL VARIETIES OF COTTON FIBERS

SUMMARY

It was the purpose of this study to determine independently the effects of (1) scouring and (2) moisture conditioning on the rate and degree of subsequent partial acetylation of six different varieties of cotton fibers chosen to represent a wide range of physical properties. The fibrous structure was maintained throughout the experimentation while acetyl contents ranging from three to twenty six per cent were obtained.

Four methods of scouring ranging from a mild scour using no caustic to a more severe scour using five per cent sodium hydroxide were employed. These scouring treatments served to remove some of the impurities such as cotton wax, oils, and pectin matter present in raw cotton which, when removed, would tend to increase the rate of subsequent acetylation by eliminating any "masking" of the available hydroxyl groups in cellulose as well as discouraging side reactions. The scoured samples were subsequently acetylated in the same manner as additional samples of unscoured cotton which were conditioned before acetylation in atmospheres over various sulfuric acid - water solutions corresponding to relative humidities of 15, 35, 50, 65, and 85 per cent at 70° F.

All varieties of cotton were presoaked in glacial acetic acid for 18 hours at 70° F. after first being subjected to one of the four methods of scouring or five moisture conditioning pretreatments described above, and then acetylated in a mixture of three parts of glacial acetic acid to one part of acetic anhydride, with 0.15 per cent perchloric acid on the mixture as catalyst. Acetylations were carried out at 64° F. for 30, 60, and 90 minutes. Analysis for acetyl content was made by saponification with sodium hydroxide solution and back titration with standardized hydrochloric acid solution against an unacetylated blank.

The results of this study proved conclusively that scouring prior to acetylation not only increased the rate of reaction but also minimized the differences in rate and degree of acetylation displayed by cotton fibers of different varieties. The increase in rate of reaction is most pronounced for mature cottons. The mild scour produced substantially the same effect as the more severe scours. Although the results of moisture conditioning were not as conclusive, it was established that an increase in the rate of acetylation followed increasing moisture contents in the range of relative humidities from 35 to 85 per cent. There was no appreciable difference, however, in the rate or degree of acetylation following moisture conditioning at 15 and 35 per cent relative humidity.

Further investigation in the field of pretreatments for cotton to increase the rate and degree of chemical reaction without significant

degradation is recommended, particularly in view of the many additional valuable properties which may be imparted to natural fibers by chemical modification.

CHAPTER I

INTRODUCTION

The chemical modification of natural textile fibers provides a means of imparting specific and desirable properties to these fibers while retaining many of the valuable characteristics normally associated with them. Cotton fibers substituted chemically by partial acetylation exhibit high resistance to mildew and microbiological rotting; resistance to the effects of high temperatures or continued heat; low moisture regain and high electrical resistance as compared with untreated cotton; and resistance to coloring with many direct dyes.[1] Furthermore, partially acetylated cotton does not change in appearance or in general textile qualities.

The Problem

Statement of the Problem.--It was the purpose of this investigation to determine independently the effects of (1) scouring and (2) moisture conditioning on the rate and degree of subsequent partial acetylation of six different varieties of cotton fibers. The fibrous structure was maintained throughout the experimentation while acetyl contents ranging from three to twenty six per cent were obtained.

Importance of the Study.--Although numerous and varied investigations have been conducted and reported in regard to the more complete

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[1] A. S. Cooper, S. T. Voorhies, E. M. Buras, and C. F. Goldthwait, "Partial Acetylation of Cotton," Textile Industries, 116:97, January 1952.

acetylations required in the preparation of cellulose acetate solutions, relatively little has been done in the field of partial acetylation of cotton in which the fibrous state is maintained. Since prolonged acetylation reactions degrade cotton cellulose without direct relationship to the degree of acetylation obtained,[2] and in order to make the process commercially feasible and economic, partial acetylation should be accomplished as rapidly as is possible consistent with control of the degree of acetylation.[3] Further, different varieties of cotton exhibit a considerable divergence in rate and degree of acetylation under identical reaction conditions.[4] By appropriate treatment of the cotton fibers prior to acetylation the rate of the reaction might be increased, thus reducing degradation, and the reactivity of the different varieties of cotton might be leveled to produce substantially the same degree of acetylation in each variety under identical reaction conditions. Accordingly, the work described herein was undertaken to determine the effects of prescouring and moisture preconditioning on the rate and degree of acetylation.

Method of Approach

Selection of the Cotton Fibers.--The six varieties of cotton fibers chosen for experimentation were selected to represent differences with respect

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[2]E. Heuser, "Cellulose: Acetylation in Phosphoric Acid Solution," Industrial and Engineering Chemistry, 40:1503, 1948.

[3]Walter M. Scott, "Some Recent Developments in Cotton Research at Southern Regional Research Laboratories," Textile Research Journal, 19: 440, 1949.

[4]S. Y. Poon, A Study of the Rate and Degree of Acetylation of Different Varieties of Cotton Fibers, Masters Thesis, Georgia Institute of Technology, 1952.

to variety and area of growth, and such properties as maturity, fineness, and crystallite orientation as measured by x-ray angle.

Properties Determined Prior to Treatment.--All fiber properties were determined at standard condition. The term "standard condition" refers to the condition of the cotton when in moisture equilibrium with a standard atmosphere having a relative humidity of 65 per cent at 70° F. The following properties of the raw cottons were determined before treatment:

1. Fiber strength.
2. Fiber fineness.
3. Moisture content.
4. Fludity.*
5. Maturity.**
6. Crystallite orientation.**

Selection of Scouring Conditions Employed.--Four different scouring treatments were used ranging from a mild scour using no caustic to a moderately severe scour using five per cent caustic. Detergent assistants of both the anionic and nonionic types were used. All scouring was done at the boil. The duration of the treatments ranged from thirty to ninety minutes.

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*These results were furnished by the Chemistry Department of the Research Division of the West Point Mfg. Co., West Point, Georgia.

**These results were furnished by the Southern Regional Research Laboratories, New Orleans, Louisiana.

Properties Determined after Scouring.--The following properties were determined at standard condition after scouring for all cottons used:

1. Fiber strength.
2. Fiber fineness.
3. Moisture content.
4. Fluidity.

Moisture Conditions Employed.--All scoured cottons were brought to standard condition before acetylation. The unscoured cottons used for the study of moisture preconditioning effects were conditioned before acetylation to moisture contents corresponding to a range of atmospheric conditions as follows:

1. 15 per cent relative humidity at 70° F.
2. 35 per cent relative humidity at 70° F.
3. 50 per cent relative humidity at 70° F.
4. 65 per cent relative humidity at 70° F. (Standard condition)
5. 85 per cent relative humidity at 70° F.

Presoaking Conditions.--Both the scoured and moisture conditioned cottons were presoaked in glacial acetic acid for 18 hours at 70° F. immediately prior to acetylation.

Acetylation Conditions.--Acetylations were carried out in a modified one pound package dyeing machine with open circulation of the acetylating mixture. Each selected cotton subjected to each different pretreatment was acetylated for 30, 60, and 90 minutes at a constant temperature of

64° F. The acetylation mixture consisted of three parts of glacial acetic acid to one part of acetic anhydride, with 0.15 per cent of 60 per cent perchloric acid on the mixture as catalyst. All water was excluded from the reaction chamber.

Properties Determined after Acetylation.--The following properties were determined for all acetylated cottons:

1. Fiber strength.
2. Fiber fineness.
3. Moisture content.
4. Per cent acetyl content.

CHAPTER II

THEORETICAL CONSIDERATIONS

The Effects of Scouring.--The natural impurities in raw cotton consist chiefly of pectin matter, cotton wax, albuminous matter and inorganic salts.[5] The amount of wax in native cotton is usually 0.5 per cent, but slight variations will be found according to the type of cotton. The wax appears to serve as a protective layer lying mostly in the cuticle on the outside of the fiber, although at least part is located inside the fiber or bound to the cellulose. The wax resists wetting, but may be removed by boiling in dilute alkali. The greater portion of the impurities present in raw cotton, however, consist of pectin matter which may also be removed by scouring with dilute alkali. A typical analysis of raw American cotton in the conditioned state shows constituents as follows:[6]

Cellulose.	91 per cent
Wax and oil.	0.35 per cent
Pectins.	0.53 per cent
Mineral matter (as ash). .	0.12 per cent
Water.	8.0 per cent

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[5] M. Plunguian, Cellulose Chemistry, Brooklyn: Chemical Publishing Co., Inc., 1943, p. 22.

[6] J. T. Marsh and F. C. Wood, An Introduction to the Chemistry of Cellulose, London: Chapman and Hall, Ltd., 1938, p. 11.

It would appear from the above data that the wax, oils, and pectin matter would be most likely to inhibit chemical reaction of the raw cotton, and that if these impurities could be removed the rate of reaction would be increased. The waxes and fatty constituents might serve to "mask" the hydroxyl groups and thus slow the reaction. Marsh has shown that the action of boiling sodium hydroxide solutions serve to saponify the waxes and fats, and that the use of detergent assistants emulsify the impurities and maintain them in a dispersed state during the scouring and washing.[7] Thus, the more complete or severe the scour up to the point where all impurities are removed, the faster the rate of subsequent acetylation. This effect should be particularly pronounced in the range of partial acetylation under investigation.

The Effect of Moisture in the Cotton.--The swelling effect of moisture on cotton fibers is of particular interest in this study. Based on the discussion of the factors affecting acetylation which follows, increased swelling results in an increase in the rate of the reaction. Within the range of humidity conditions employed in this investigation, the cross-sectional swelling of cotton fibers has been shown to be nearly linearly related to the relative humidity of the atmosphere in which they are allowed to come to moisture equilibrium. Cotton fibers at 15 per cent relative humidity show a cross-sectional swelling of 4.2 per cent as compared to the bone dry diameter, while fibers conditioned at

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[7] J. T. Marsh, An Introduction to Textile Bleaching, New York: John Wiley and Sons, Inc., 1948, pp. 185-196.

85 per cent relative humidity swell 11.75 per cent. At 35, 50, and 65 per cent relative humidities, the fibers swell 7.1, 8.9, and 9.9 per cent, respectively.[8]

The moisture content of the cotton before acetylation may also have an effect upon the ultimate strength of the acetylated fiber. Investigation has shown that films prepared from acetylated fibers which had been swollen in water prior to acetylation exhibited greater tensile strength with increasing water content, the maximum being reached at a moisture content of 20 per cent.[9]

The Effect of Acetic Acid Presoak.--Acetic acid acts as a preswelling agent in the same way as water. Cotton which has been preswollen with 200 per cent of its weight in glacial acetic acid reacted about ten times faster than without pretreatment.[10] It is assumed, however, that there is a difference in the degree of swelling produced by water and acetic acid, and that the moisture content of the cotton when placed in the acetic acid presoak bath will influence the rate of acetylation. The swelling effect of water is felt to be superior to that of acetic acid, and therefore the moisture content is the predominant factor in

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[8] F. F. Morehead, "A Method for Studying the Effect of Humidity on the Cross-sectional Swelling of Some Common Fibers," Textile Research Journal, 22: 535, 1952.

[9] E. Elod, H. Schmid-Bielenberg, and L. Thoria, "Cellulose Fibers: Acetylation", Agnew Chem. 47: 465, 1934 through Journal of the Textile Institute, 25: A420, 1934.

[10] E. Heuser, The Chemistry of Cellulose, New York: John Wiley and Sons, Inc., 1944, p. 245.

regard to swelling. The acetic acid may merely maintain the degree of swelling previously produced by the water while replacing it in the capillaries of the fiber, although some additional "activation" may occur. Since it is necessary to exclude all water from the acetylation reaction due to the presence of acetic anhydride and perchloric acid, the hypothesis is advanced that the primary function of the acetic acid presoak is to replace the water present in the cotton, or at least to dilute it to an extremely low concentration in the solution.

Factors Affecting Acetylation.--Chemically, the reactions of cellulose are chiefly those which are characteristic of organic compounds having an abudance of hydroxyl groups. An examination of the cellulose formula shows three hydroxyl groups free to react in each glucose residue. By virtue of their positions in the residue and their primary and secondary natures, it might be expected that they would show significant differences in reactivity.[11] However, in acetylation reactions it appears that there is no difference in the functional value of the hydroxyl groups, but rather that their physical accessibility in the fibrous structure determines their preference for reaction.[12]

Thus the major factors affecting the rate of reaction are the degree of crystallinity and the orientation of the crystalline structure. Where the cellulose chains of cotton fibers are densely packed and regularly oriented, chemical reaction is slow. The portions of the fiber

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[11] Ibid. p. 40.

[12] E. Heuser, "Factors Which Influence the Kinetics of Cellulose Reactions," Textile Research Journal, 20: 834, 1950.

with an expanded structure permit penetration of the reagents and thus are more reactive. Therefore it may be said that the crystalline portions react more slowly than the amorphous regions. There is no clearly defined border between these regions, but a transitional region exists between them which reacts to an intermediate degree.[13] This view is supported by Ward[14] who states that the amorphous regions as well as the surfaces of the crystalline regions are readily accessible for chemical reactions. Since x-ray studies indicate the crystalline fraction of cotton cellulose to be approximately 70 per cent,[15] somewhat over 30 per cent of the fiber is readily accessible for acetylation.

The degree of swelling of the submicroscopic structure of the fiber as a factor affecting acetylation has been discussed in regard to moisture content and acetic acid presoaking. The swelling of the fiber promotes the rate of diffusion of the acetylating agent by enlarging the intermicellar spaces. Therefore, any compound or pretreatment which enlarges the lattice spacings and permits faster diffusion into the crystallite will increase the reaction rate.

The reagents employed in acetylation are, of course, factors which affect the reaction. Acetic acid alone reacts reluctantly with cotton. Experiments performed on surgical cotton using boiling acetic

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[13] A. Meller, "Fibrous Cellulose: Reactivity," Journal of Polymer Science, 4: 619, 1949.

[14] K. Ward, "Crystallinity of Cellulose and its Significance for the Fiber Properties," Textile Research Journal, 20: 363-372, 1950.

[15] P. H. Hermans and A. Weidinger, "X-Ray Studies on the Crystallinity of Cellulose," Journal of Polymer Science, 4: 135-144, 1949.

acid as the acetylating agent produced an acetyl content of only 6.4 per cent after 500 hours of reaction. Acetic anhydride is the most efficient acetylating agent, although acetyl chloride or ketene may be used successfully.[16] The function of the "catalyst" in the reaction is not clearly understood, although Heuser considers the chief function to be swelling, and therefore does not attribute a true catalytic effect to either sulfuric or perchloric acid.[17] Perchloric acid is the most powerful catalyst for the acetylation of cellulose, producing a higher degree of acetylation in a much shorter time than sulfuric acid.[18] It is undoubtedly for this reason that perchloric acid is preferred in partial acetylation. Recent experiments with acetic acid/acetic anhydride systems clarify the function of the catalyst. The exhibition of "super acidity" by the system in the presence of strong acids such as perchloric is accounted for by postulating the existence of highly acidic acetyl ions in the solution which would take part in an esterification reaction with the hydroxyl groups of cellulose much more readily than if in a less active state.[19]

Probable Course of the Acetylation Reaction.--The heterogeneous nature of the acetylation reaction is due both to the fiber and micellar structure

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[16]E. Heuser, The Chemistry of Cellulose, New York, John Wiley and Sons, Inc., 1944, p. 227, 281-284.

[17]Ibid. p. 231.

[18]Ibid. p. 240.

[19]H. A. E. Mackenzie, "Acetic Acid/Acetic Anhydride System: Properties," Transactions of the Faraday Society, 44: 159, 1948.

of cotton. In the course of the reaction the reagents penetrate the fiber, attacking first the amorphous regions and the surfaces of the crystalline regions. The reaction then proceeds inwards producing between the unreacted interior of the highly oriented crystalline regions and the completely reacted exterior surface a partially reacted area.[20] Therefore, all three forms of cellulose acetate exist in the fibers of partially acetylated cotton; at least part of the amorphous regions and micelle or chain bundle surfaces are converted to the triacetate, while the monoacetate and diacetate forms exist in varying degree towards the interior of the crystallites. The hydroxyl groups in all regions behave toward the acetylating agents as though they were chemically equal.

The action of the catalyst at various stages of acetylation has been examined to show that the perchloric acid first forms an addition complex with the cotton. As acetylation proceeds, this complex is slowly broken down by the introduction of acetyl groups, and at the same time small amounts of perchlorate ester groups are formed. Theoretically the triacetate portions would contain no perchloric acid, although the partially acetylated areas may contain perchlorates in the form of the initial complex or as an ester.[21]

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[20] W. A. Sisson, "X-Ray Diffraction Behavior of Cellulose Derivatives," Industrial and Engineering Chemistry, 30: 530, 1938.

[21] G. Petitpas, "The Perchloric Acid Catalysis of the Acetylation of Cellulose," Mem services chim etat, 31: 178-186, 1944, through Chem. Abst., 40: 7613, 1946.

CHAPTER III

INSTRUMENTATION AND EQUIPMENT

Acetylation Machine.--A Morton one pound capacity package or cheese dyeing machine was modified for acetylation by placing a single pass stainless steel cooling coil in series with the reaction or dyeing chamber. A by-pass valve was installed so that all, none, or a regulated quantity of the acetylation mixture could be circulated through the cooling coil to maintain constant temperature. The machine was constructed of stainless steel throughout, and a sealed centrifugal pump provided circulation. The cooling coil was mounted in a galvanized sheet metal tank which was filled with ice and water for cooling during acetylation. The necessary additional piping and the cooling coil increased the liquid capacity of the machine from approximately nine liters to 16.8 liters. Photographs of the modified machine are shown in Figures 24 and 25 in the Appendix.

Sheffield Micronaire Model No. 60600.

Pressley Fiber Strength Tester.

Brabender Moisture Tester.

Humidifying Chamber.--Standard ten inch desiccators with sulfuric acid-water solutions were used as humidifying chambers.

Laboratory Cutting Mill, Wiley, Intermediate Model.

Titration Apparatus.--Standard laboratory equipment such as burettes, Erlenmeyer flasks, and analytical balances were used in the titration procedures.

Hot Air Circulating Drying Oven.

Fletcher Centrifugal Extractor, 1400 RPM.

List of Reagents Employed.--

Duponel RA. (E.I. DuPont de Nemours Co., Inc.)

Merpol C. (E. I. DuPont de Nemours Co., Inc.)

Triton X-100. (Rohm and Haas Co.)

Glacial Acetic Acid, Tech.

Acetic Anhydride, Tech.

Perchloric Acid 60%.

Chlorantine Fast Blue 3 RLL. (Ciba Co.)

Celliton Fast Yellow RRA. (General Dyestuff Corp.)

Standard Chemical Laboratory Reagents.

CHAPTER IV

EXPERIMENTAL PROCEDURE

Testing of Fibers Before Treatment.

Fiber Strength.--Samples of each of the raw cottons selected for experimentation were tested at standard condition prior to treatment for strength index according to the method described in A.S.T.M. Standards on Textile Materials D 414-49T. The Pressley Cotton Fiber Strength Tester was used in making these determinations.

Fiber Fineness.--The fiber fineness of each sample of raw cotton was determined at standard condition prior to treatment by means of the Sheffield Micronaire, and in accordance with the procedure recommended by the manufacturer.[22]

Moisture Content.--The percent moisture of each variety of raw cotton under experimentation was determined at standard condition using the Brabender Moisture Tester. Ten gram samples of each variety of cotton were accurately weighed on the balance furnished with the tester. Before use this balance was calibrated against a standard laboratory analytical balance. A tolerance of plus or minus 25 milligrams was allowed in initial weighings. The weighed samples were then placed in perforated aluminum drying cans in the oven of the moisture tester and maintained

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[22]Sheffield Corporation, "Instructions for Use of the Sheffield Micronaire," (Dayton, Ohio: Sheffield Corp.)

under forced air circulation at a temperature of 110 degrees Centigrade until constant weight was reached. The per cent moisture was then read directly off the scale of the moisture tester. This scale is calibrated on the basis of a ten gram conditioned sample in accordance with the following formula:

$$M = \frac{100(a-b)}{a}$$

where

a = weight of conditioned sample (ten grams)

b = weight of sample after drying

M = per cent moisture

Fluidity.--The fluidity by dispersion in cupriethylene diamine hydroxide of each of the untreated cottons was determined by the Research Division of the West Point Manufacturing Company, West Point, Alabama in accordance with A.S.T.M. D-539-48T, Method B. Two determinations were made on each sample, and the average taken as an indication of the degree of polymerization.

Maturity.--The per cent maturity of each variety of raw cotton was determined by swelling in 18 per cent sodium hydroxide according to the method described in A.S.T.M. D-444-49T. These determinations were furnished by the Southern Regional Research Laboratory.

Crystallite Orientation.--The crystallite orientation as measured by x-ray angle of each variety of raw cotton was also furnished by the Southern Regional Research Laboratory.

Scouring Treatments

General Procedure.--The one pound package dyeing machine subsequently used for acetylation also was used for all scouring treatments. Seventy five grams of each of the six varieties of cotton were placed in six separate layers in the cylindrical reaction chamber of the machine. Each layer was separated from adjacent layers by circular stainless steel screens which allowed free passage of the scouring liquor, but prevented admixture of the different cottons. In this manner each variety of cotton was subjected to an identical scouring treatment. In addition, a sufficient amount of cotton was scoured at one time to provide all the samples required for subsequent acetylations.

Warm water (136° F.) was first added to the reservior of the machine to a total volume of nine liters. The scouring agents were then added and the temperature raised to the boil in one half an hour. The timing of each scouring treatment commenced after boiling started. At the expiration of each scouring treatment the cotton was given a ten minute warm water rinse followed by a twenty minute cold water running rinse. Excess water was removed from the samples in the centrifugal extractor and they were then dried in a hot air circulating oven at approximately 140° F.

Scouring Conditions Employed.--Listed below are the times, temperatures, and reagents used for each of the four methods of scouring employed. Method I is a mild scour using no caustic as recommended by a representative of E.I. DuPont de Nemours and Company, Incorporated. Method II is

a severe scour using a solvent containing detergent and was also recommended by a representative of the DuPont Company. Method III was recommended in a technical bulletin of the Rohm and Hass Company and employs a non-ionic type detergent assistant. Method IV uses the highest percentage of caustic, assisted by a neutral oleate soap, and was taken from a general discussion of scouring procedures by Marsh.[23]

Method I

1.0 per cent Duponol RA (Long chain alcohol sulfate)
1.5 per cent tetrasodium pyrophosphate
Scour cotton 30 minutes at the boil.
Rinse in warm water and dry.

Method II

2.0 per cent sodium hydroxide
1.0 per cent Merpol C (Higher alcohol sulfate containing solvents)
0.5 per cent Duponol RA
0.5 per cent tetrasodium pyrophosphate
Scour cotton 60 minutes at the boil.
Rinse in warm water and dry.

Method III

3.0 per cent sodium hydroxide
0.1 per cent Triton X-100 (Iso-octyl phenyl ether of polyethylene glycol)
0.3 per cent tetrasodium pyrophosphate
Scour cotton 60 minutes at the boil.
Rinse in warm water and dry.

Method IV

5.0 per cent sodium hydroxide
2.0 per cent soap (neutral oleate)
Scour cotton 90 minutes at the boil. Rinse and dry.

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[23] J. T. Marsh, An Introduction to Textile Bleaching, New York: John Wiley & Sons, Inc., 1948, pp. 185-196.

All per centages are based on the weight of the cotton, and all scourings were made in a 20:1 bath.

Testing of Fibers after Scouring

Fiber Strength.--Samples of each of the scoured cottons were tested at standard condition for strength index in a manner similar to that described under testing before treatment. A comparison of the strength index before and after scouring gives an indication of loss of strength caused by the scouring treatments, and permits evaluation in this respect of one scouring method against another.

Fiber Fineness.--The fiber fineness of each variety of scoured cotton was also determined in a manner similar to that described under testing of fibers before treatment, thus allowing a comparison of the swelling effects, if any, of the various methods of scouring.

Moisture Content.--The moisture content of each scoured cotton was determined at standard condition in the same manner as described under testing before treatment.

Fluidity.--As in the case of the untreated cottons, two determinations of fluidity by dispersion in cupriethylene diamine hydroxide were made on each sample of scoured cotton. The average values of these determinations were used as sensitive measures of the degradation of the cottons due to scouring, supplementary to the fiber strength determinations described above.

Moisture Conditioning Prior to Acetylation

Apparatus and Method Used.--All scoured samples were brought to standard condition before presoaking in glacial acetic acid preparatory to acetylation. For this purpose a laboratory was available which was maintained at a constant temperature of 70° F. and a constant relative humidity of 65 per cent by mechanical air conditioning equipment.

To determine the effects of various moisture conditions on subsequent partial acetylation, additional samples of each of the six varieties of raw cotton were conditioned to five different moisture contents corresponding to atmospheric conditions of 15, 35, 50, 65, and 85 per cent relative humidity and 70 F. In order to produce the desired atmospheric conditions, standard ten inch desiccators containing various sulfuric acid - water solutions were used. Six 20 gram samples of cotton were conditioned at one time in each desiccator, each sample representing one of the varieties of cotton chosen for the study. In order to facilitate rapid transfer of the samples from the humidifying chamber to the presoak container, thus avoiding an appreciable change in the moisture content of the conditioned cottons, these six samples were arranged in horizontal layers around a vertical wooden spindle with circular stainless steel screens used to separate the layers. The cotton samples were then placed in the humidifying chamber and allowed to condition for a minimum of 24 hours to assure that moisture equilibrium was reached.

Preparation of Humidity Solutions.--In order to produce an atmosphere corresponding to the desired relative humidity conditions at 70 F., sulfuric acid solutions were prepared for use in the humidifying chambers in accordance with Table 1. A total volume of 400 milliliters of each solution was prepared using 1.834 specific gravity sulfuric acid and water. The solutions were cooled to 70° F. and adjusted to the proper specific gravity using a standard laboratory hydrometer.

Table 1. Relative Humidities above Sulfuric Acid Solutions[24]

Relative Humidity per cent	Concentration of Sulfuric Acid % Weight/Volume	Specific Gravity	Degrees Baume
15	61.0	1.51	49.2
35	50.3	1.40	41.6
50	43.9	1.34	36.9
85	22.5	1.16	20.2

The samples to be conditioned at 65 per cent relative humidity were allowed to remain the standard atmosphere of the laboratory rather than being placed over an appropriate sulfuric acid solution. The humidifying chambers also remained in this laboratory to assure a constant temperature of 70 F.

[24] J. H. Skinkle, Textile Testing, Brooklyn: Chemical Publishing Co., Inc., 1949, p. 18.

Determination of Moisture Content after Conditioning.--In order to determine the approximate moisture contents after conditioning as described above, ten gram samples of each variety of cotton were placed in the perforated aluminum cans of the Brabender Moisture Tester and allowed to come to moisture equilibrium in the various humidifying chambers. They were then removed from the chambers, immediately placed in the oven of the moisture tester, weighed, dried to constant weight, and reweighed. The moisture contents were then calculated according to the formula shown in the section describing the testing for moisture content before treatment. (p. 15)

Acetylation

Presoaking Procedure.--All samples were presoaked in glacial acetic acid for 18 hours at 70 F. immediately prior to acetylation.

The previously scoured samples were brought to standard condition before presoaking. Twelve 20 gram samples were presoaked at one time, having been previously arranged around the stainless steel spindle used in the acetylating machine and separated from each other by circular stainless steel screens. In this manner six varieties of cotton subjected to scouring method I were presoaked in a common bath with six samples which had been scoured according to method II. Samples scoured by methods III and IV were similarly combined.

Six 20 gram samples of the cottons which had not been scoured but which had been brought to moisture equilibrium with atmospheres of various relative humidities were presoaked at one time on the same wooden spindles

used in the humidifying chambers. Care was taken to effect a rapid transfer of these samples from the humidifying chambers to the presoak baths so as to minimize changes in moisture content due to exposure to the standard atmosphere of the laboratory.

Covered stainless steel beakers were used to hold the presoak bath. The inside diameters of these beakers corresponded to the outside diameters of the stainless steel separatory screens and the inside diameter of the reaction chamber of the acetylation machine. The beakers were therefore 6-3/8 inches in diameter, 7-1/2 inches in height, and held 3.6 liters of glacial acetic acid when filled so as to cover completely all the samples being presoaked.

Preparation of the Acetylation Mixture.--The acetylation mixture was prepared in a glass carboy of approximately 20 liter capacity. A mixture containing 11.6 liters of glacial acetic acid and 4.2 liters of acetic anhydride was cooled to 50 F. in the carboy by placing it in a water-ice bath. One liter of glacial acetic acid used to dilute 25.5 milliliters of 60 per cent perchloric acid was then added and the mixture again cooled to 50 F. The mixture was then ready for use in the acetylating machine. In summary, the total amounts of materials used in each acetylation are:

12.6 liters of glacial acetic acid,
4.2 liters of acetic anhydride, and
25.5 milliliters of 60 per cent perchloric acid.

Acetylation Procedure.--Prior to acetylation all water was removed from the acetylation machine by circulating a previously used acetylation

mixture through all parts of the system including the cooling coil, reaction chamber, and reservior. The machine was then drained, water and ice was added to the tank surrounding the cooling coil, and the valves of the machine were set for open circulation through the reservior. The by-pass valve in the cooling coil system was closed so that all of the circulating acetylation mixture would pass through the coil. The cotton to be acetylated was removed from the presoak bath and placed in the reaction chamber of the machine. In the case of the scoured cottons twelve samples had already been loaded on the stainless steel spindle for presoaking as previously described. The unscoured moisture conditioned cottons, however, had to be transferred from the two wooden spindles used in presoaking to the single stainless steel spindle used in acetylation. Twelve samples of 20 grams each, arranged in layers and separated by stainless steel screens, were acetylated at one time in both cases. In transferring the cottons from the presoak bath to the acetylating machine excess acetic acid was partially removed by hand squeezing. The cover was then placed on the reaction chamber and sealed. The precooled acetylation mixture was poured into the reservior of the machine, the circulation pump was started, and the timing of the reaction begun. By regulating the by-pass valve in the cooling coil system the temperature was kept constant at 64 F., plus or minus 0.5 degrees. The temperature was read by means of a laboratory thermometer, the bulb of which was placed in the over-flow stream from the reaction chamber. Separate acetylations of 30, 60, and 90 minutes were performed for each variety of cotton and for each pretreatment employed.

Washing and Drying after Acetylation.--At the expiration of the required time of acetylation the circulating pump was stopped and the machine drained. A running cold water rinse under pump circulation was then given by introducing a steady flow of tap water into the reservior and adjusting the drain valve of the machine to maintain the water level in the reservior. This cold water rinse was continued for 20 minutes, or until foaming stopped. The drain valve was then closed and the flow of tap water stopped when the reservior was filled. Fifty milliliters of ammonium hydroxide were added to the water and circulated through the machine for ten minutes to neutralize any residual acidity. A second cold water running rinse was then given until the drain water was neutral to litmus paper. The samples were removed from the machine, damp dried in a centrifugal extractor, and then placed in a hot air circulating oven at 140 F. to dry.

Testing of Fibers After Acetylation

Analysis for Acetyl Content.--One to 1.5 gram samples at standard condition were handpicked from the original 20 gram samples of acetylated cotton and ground in a Wiley Mill to pass a 20 mesh screen. They were then transferred into tared 250 milliliter Erlenmeyer flasks and the gross weights accurately determined on an analytical balance. The exact net weights of the ground samples were then calculated and converted into dry weights using the per cent moisture figures obtained for each sample as described in the succeeding section on moisture content determination. Fifty milliliters of 75 per cent methyl alcohol were then added to each flask

and the flasks were heated for 15 to 20 minutes at 140 F. with stoppers loosely inserted. To each flask was then added fifty milliliters of an approximately 0.5 normal sodium hydroxide solution and they were again heated at 140 F. for one hour. The flasks were then tightly stoppered and the samples were allowed to saponify overnight. The rubber stoppers and the walls of each flask were then washed down with distilled water, and two or three drops of phenolphthalein were added for use as an indicator in titration. Standardized approximately 0.5 normal hydrochloric acid solution was used for the titrations. A 50 milliliter burette was used to measure the standardized acid, and the amount required to titrate each sample until the pink color disappeared was accurately determined. Two blanks of unacetylated cotton were included with each set of samples and carried through the same procedure. Two determinations for acetyl content were made for each sample of acetylated cotton, and the average value was accepted as correct if the two determinations were within 0.5 per cent of each other. The acetyl content was calculated using the following formula:

$$\text{Per cent acetyl} = \frac{(a-b) \times N \times 0.04302 \times 100}{d}$$

where

a = Volume of HCl in milliliters required to titrate the blank,

b = Volume of HCl required to titrate the sample,

N = Normality of HCl solution,

d = Dry weight of the sample in grams, and

0.04302 = Milliequivalent weight of the acetyl group.

Fiber Strength, Fiber Fineness, and Moisture Content.--After acetylation, samples of each cotton were tested for fiber fineness, fiber strength, and moisture content in the same manner as that described in the section on testing before acetylation.

Eveness of Acetylation.--Two to three gram samples of each acetylated cotton were entered into a dye bath at 180 to 190 F. containing 4 per cent Chlorantine Fast Blue 3 RLL, 4 per cent Celliton Fast Yellow RRA, and 5 per cent Triton X-100 as a wetting agent, calculated on the weight of the samples. The liquor ratio of the dye bath was 70:1. After dyeing for 30 minutes 50 per cent anhydrous sodium sulfate on the total sample weight was added and dyeing continued for another 30 minutes. The dyed samples were then washed well with cold water and dried in the hot air circulating oven. Since the yellow dye is an acetate dye which will not stain cotton and the blue dye is a direct cotton dye which will not stain cellulose acetate, the well acetylated portions of the fiber dye to a full shade of yellow and the unacetylated portions, if any, dye blue. Portions acetylated to an intermediate degree may appear to be green. The depth of shade indicates visually the degree of acetylation. The primary function of the dye test in this investigation, however, was to indicate the evenness of acetylation.

Table 2. Physical Properties of Cottons Selected for Experimentation

Cottons	NaOH* Maturity (per cent)	Micronaire Reading	Pressley Index	X-Ray Angle* 40%
Memphis	38	2.45	7.15	37.92
Empire Bale 92	72	3.70	7.30	34.08
Bob Shaw	88**	5.10	8.17	31.32
Stoneville 2B Bale 249290	80	3.20	8.07	31.20
Acala 1517	86	4.00	8.93	29.76
Lockett 140	92	5.67	7.51	36.18

*Data furnished by Southern Regional Research Laboratory.
**Maturity determined by Arealometer.

Table 3. A Comparison of the Fluidity Values of Cottons After Scouring

Cottons	Fluidity value in Rhes*			
	Scour I	Scour II	Scour III	Scour IV
Memphis	3.7	3.8	4.1	4.7
Empire Bale 92	3.8	3.7	3.2	4.1
Bob Shaw	3.1	3.1	3.0	3.1
Stoneville 2B	2.9	3.3	3.5	3.5
Acala 1517	3.6	3.9	3.4	3.4
Lockett 140	3.2	3.1	3.2	3.0

*Data furnished by West Point Manufacturing Company.

The Effect of Scouring on Subsequent Acetylation.--Table 4 gives the acetyl contents of the different varieties of scoured cottons after acetylation for 30, 60, and 90 minutes. These results are illustrated graphically in Figures 2 through 11, with the change in acetyl content plotted against the time of acetylation. For purposes of comparison, Figure 1 shows the change in acetyl content with time of acetylation for unscoured cottons previously brought to standard condition.

By comparing Figure 1 with Figures 2, 3, 4, and 5 it is readily apparent that scouring prior to acetylation tends to minimize the differences in the rate and degree of acetylation of the different varieties of cotton. For example, an examination of the 60 minute acetylations of the unscoured cottons reveals a variation in acetyl content of from 10.8 to 19.5 per cent for a maximum difference of 8.7 per cent, while the variation for the same period of acetylation for the scoured cottons ranged from a maximum difference of 2.4 per cent for cottons scoured by method III to 1.4 per cent for cottons scoured according to method I.

Furthermore, all scouring treatments increase the rate of subsequent acetylation for all cottons investigated. This increase in rate and therefore degree of acetylation is illustrated for each type of cotton in Figures 6 through 11, and is particularly noticeable with the more mature varieties of cotton. For example, an examination of Figure 11 reveals that although a 60 minute acetylation resulted in an acetyl content of only 10.8 per cent for unscoured Lockett 140 cotton, this same cotton when scoured before acetylation reached an acetyl

Table 4. Acetyl Contents of Cottons Partially Acetylated After Various Scouring Treatments

Cottons	Acetylated at 64 F. Per cent acetyl after acetylation for: 30 minutes	60 minutes	90 minutes
UNSCOURED:			
Memphis	14.0	19.5	23.5
Empire Bale 92	8.00	13.5	18.8
Bob Shaw	5.95	10.6	15.6
Stoneville 2B	9.37	15.1	19.2
Acala 1517	7.95	14.0	20.3
Lockett 140	6.32	10.8	13.5
SCOUR I:			
Memphis	18.3	20.8	26.1
Empire Bale 92	16.3	20.9	24.2
Bob Shaw	16.1	19.7	23.8
Stoneville 2B	16.2	20.4	23.6
Acala 1517	15.9	21.1	25.0
Lockett 140	15.6	19.8	24.4
SCOUR II:			
Memphis	17.9	21.2	26.1
Empire Bale 92	16.4	20.6	25.3
Bob Shaw	14.9	20.0	24.8
Stoneville 2B	16.3	20.2	25.0
Acala 1517	17.9	21.5	25.0
Lockett 140	15.8	20.4	25.6

Table 4. (Continued) Acetyl Contents of Cottons Partially Acetylated After Various Scouring Treatments

Cottons	Acetylated at 64 F. Per cent acetyl after acetylation for: 30 minutes	60 minutes	90 minutes
SCOUR III:			
Memphis	19.2	23.2	24.6
Empire Bale 92	17.0	22.0	23.7
Bob Shaw	16.0	21.5	23.0
Stoneville 2B	16.8	22.5	23.8
Acala 1517	17.0	22.7	24.6
Lockett 140	15.4	20.8	23.8
SCOUR IV:			
Memphis	18.3	23.7	25.4
Empire Bale 92	16.7	22.9	24.5
Bob Shaw	16.3	21.5	24.2
Stoneville 2B	17.6	22.5	24.8
Acala 1517	18.0	23.3	26.0
Lockett 140	16.3	21.9	24.8

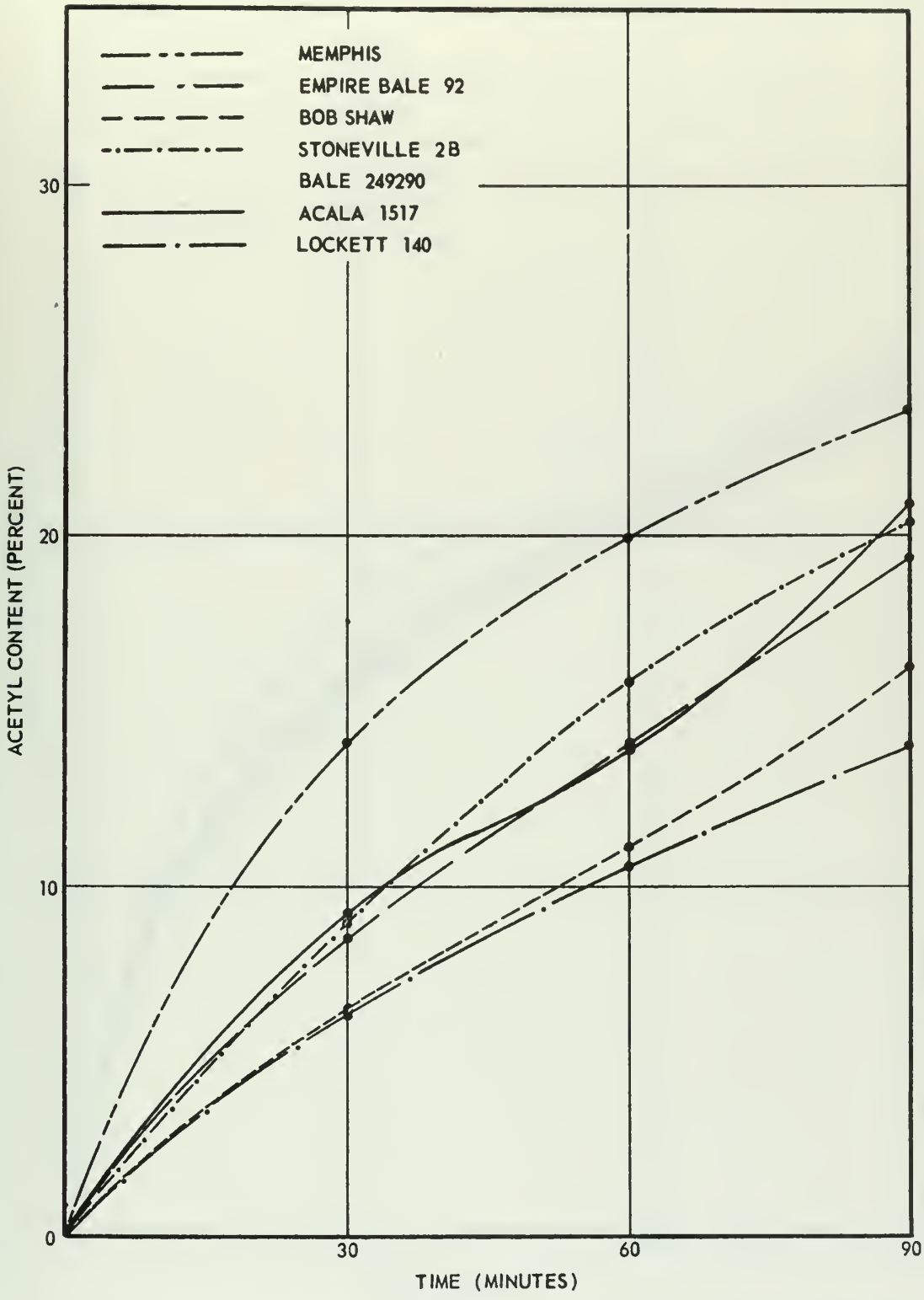

Figure 1. The Change in Acetyl Content With Time of Acetylation for Unscoured Cottons.

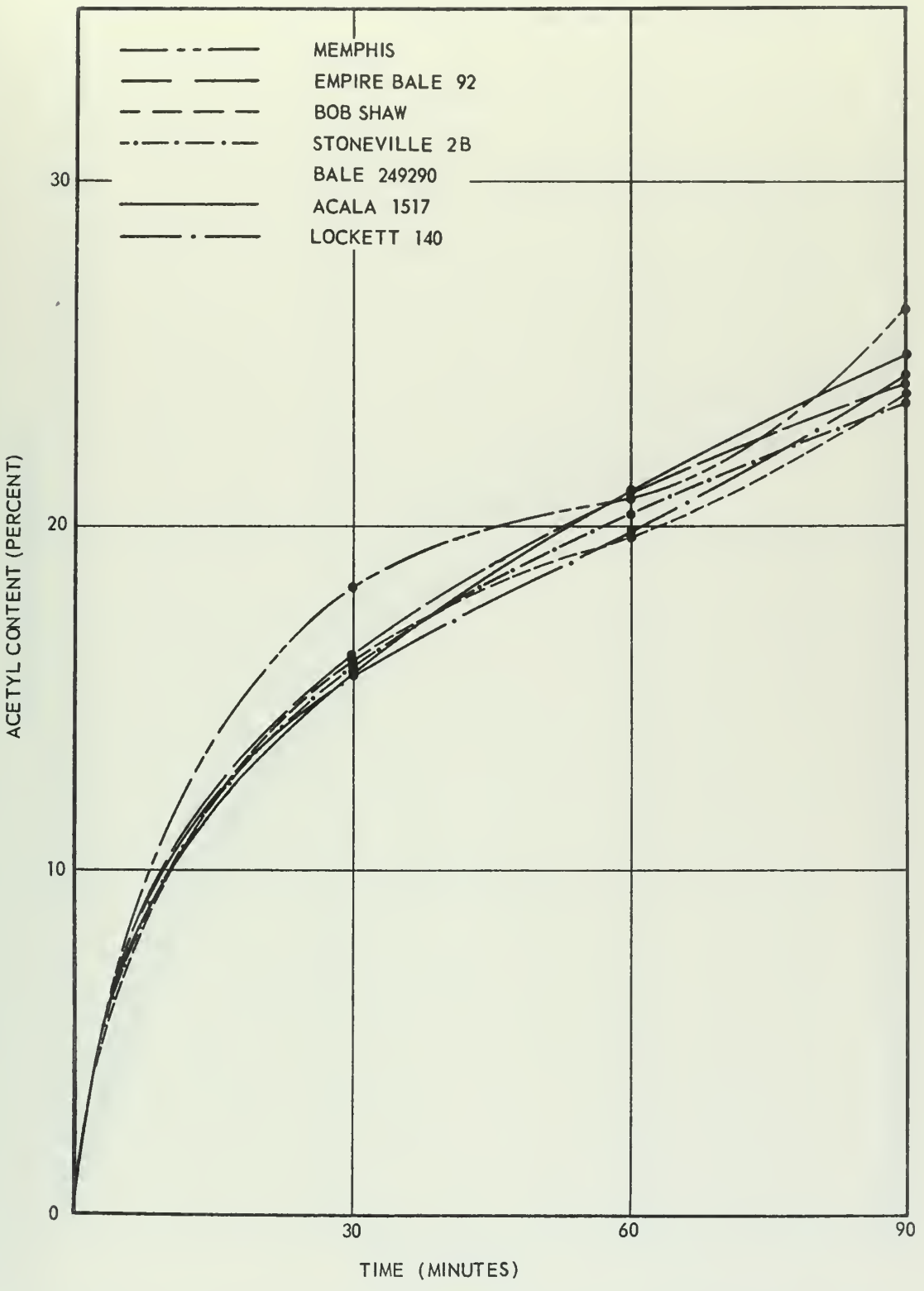

Figure 2. The Change in Acetyl Content With Time of Acetylation for Cottons Scoured by Method I.

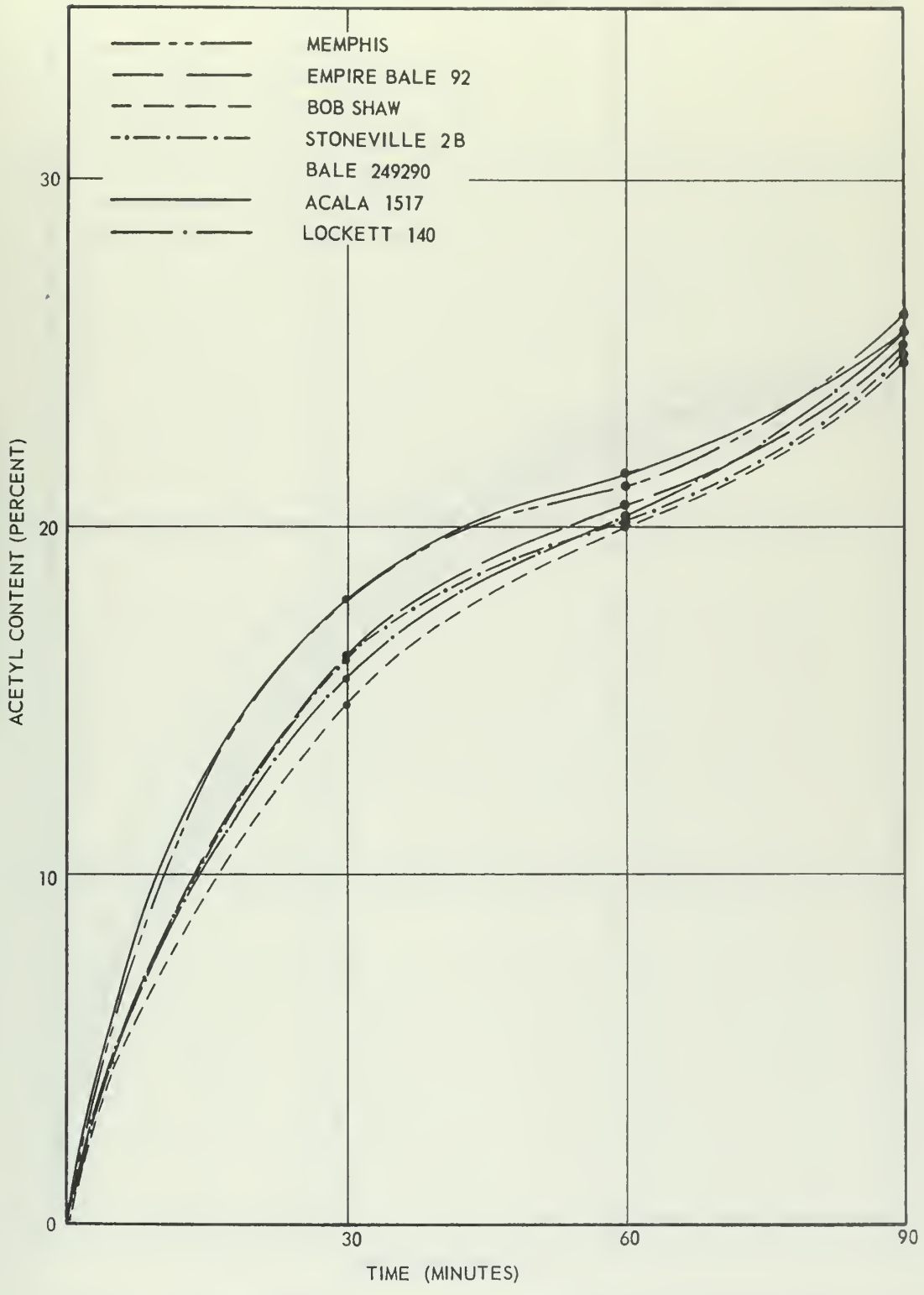

Figure 3. The Change in Acetyl Content With Time of Acetylation for Cottons Scoured by Method II.

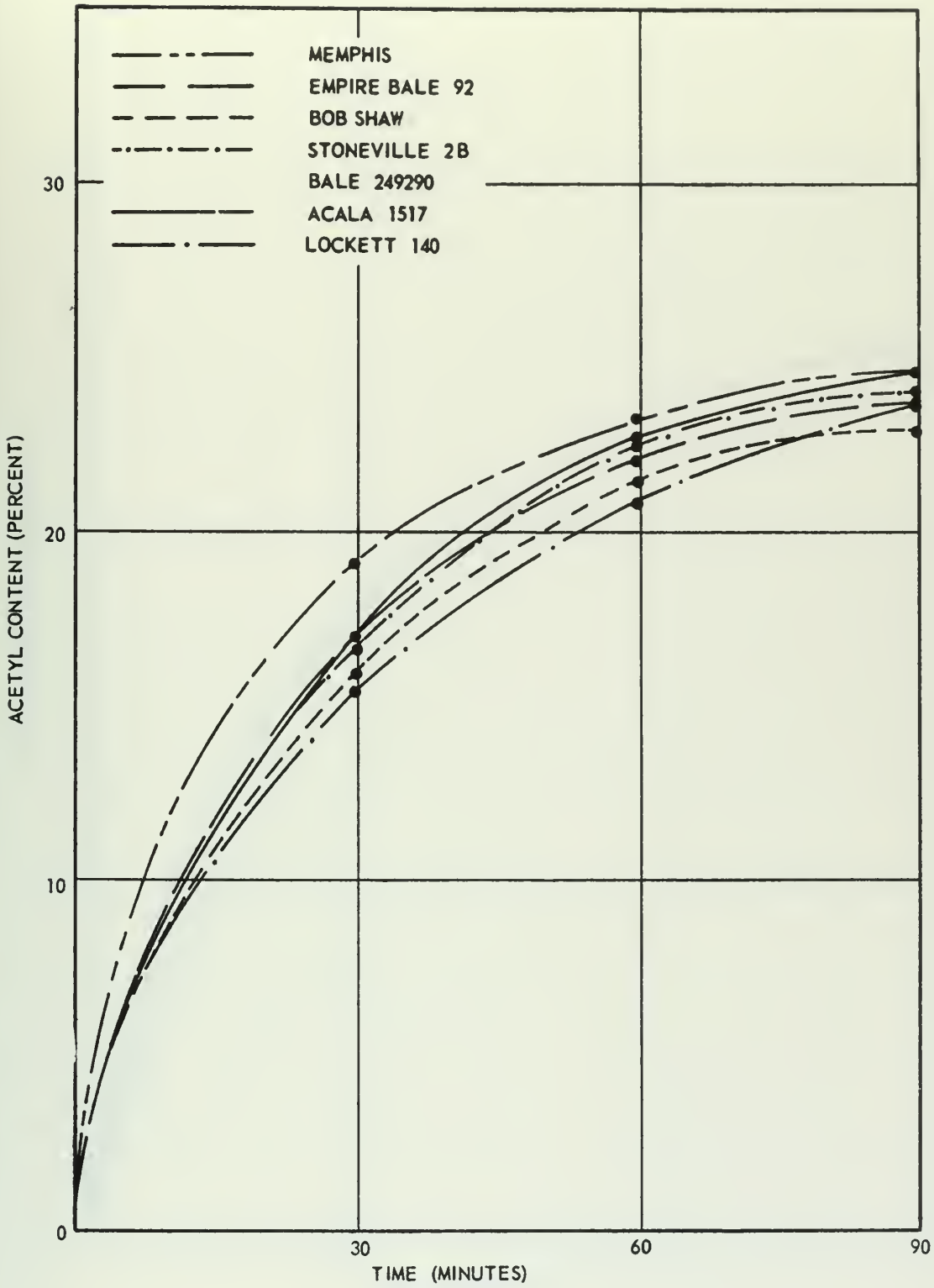

Figure 4. The Change in Acetyl Content With Time of Acetylation for Cottons Scoured by Method III.

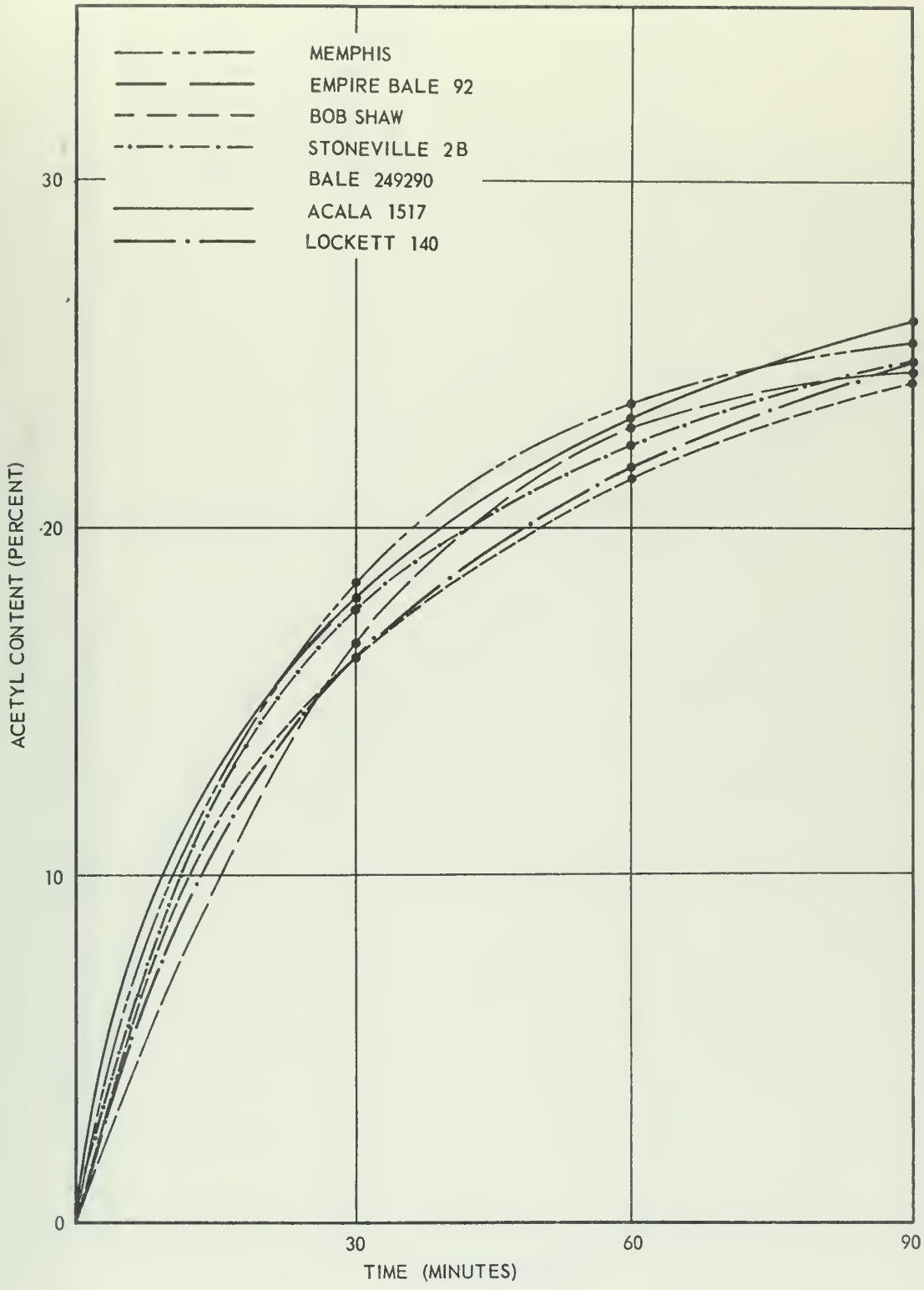

Figure 5. The Change in Acetyl Content With Time of Acetylation for Cottons Scoured by Method IV.

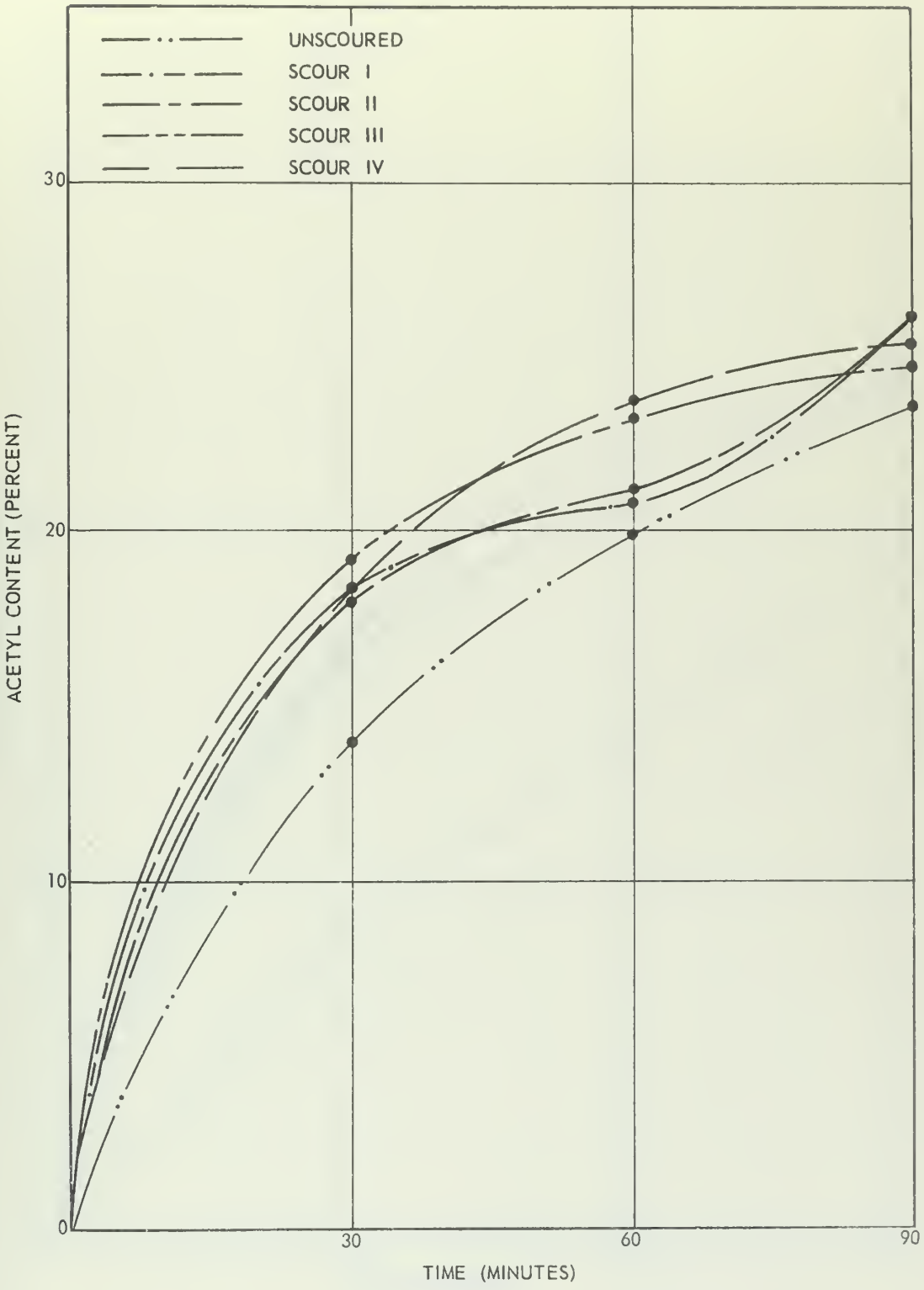

Figure 6. The Change of Acetyl Content With Time of Acetylation for Memphis Cotton Unscoured and Scoured by Four Methods.

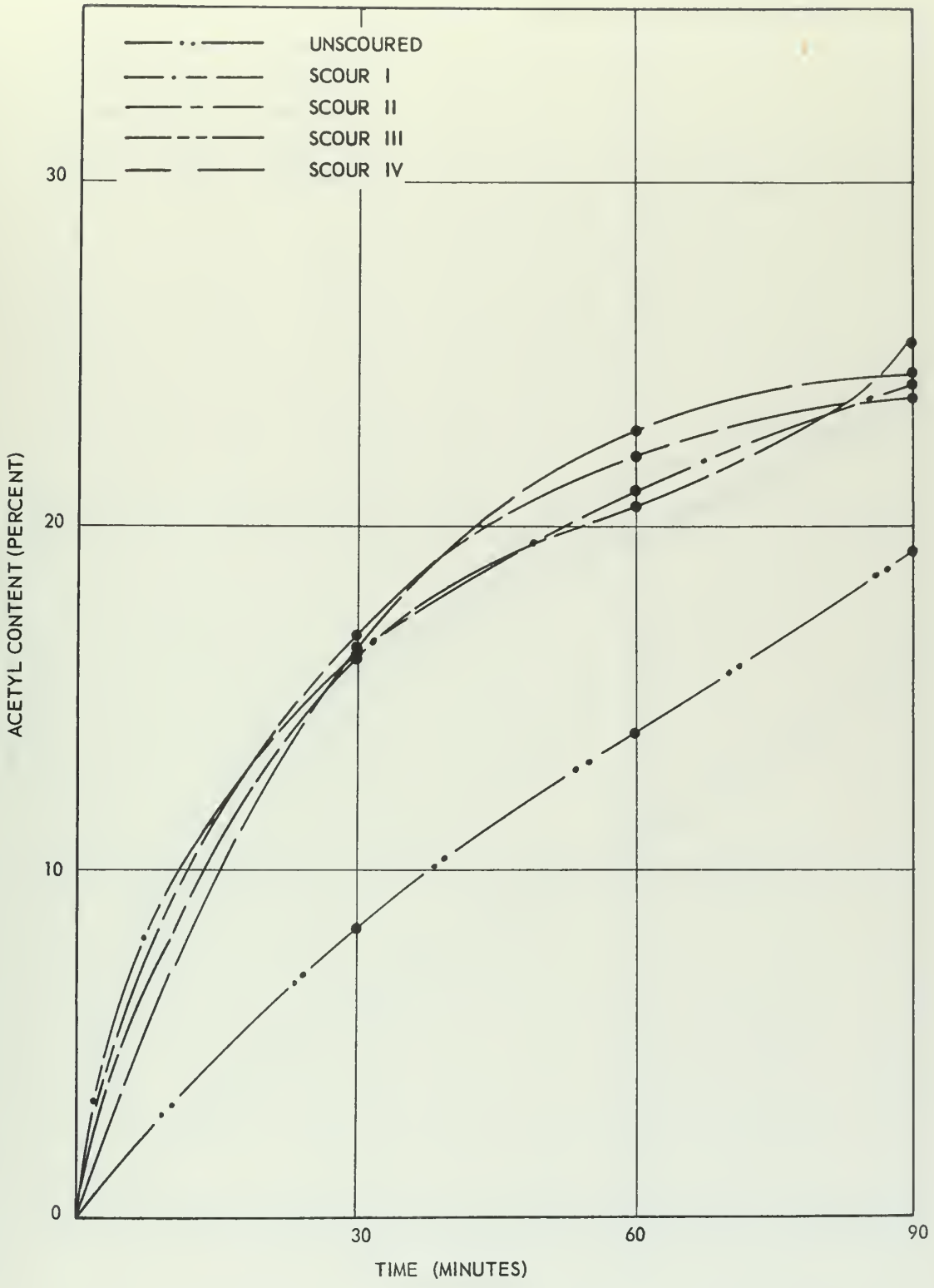

Figure 7. The Change of Acetyl Content With Time of Acetylation for Empire Bale 92 Cotton Unscoured and Scoured by Four Methods.

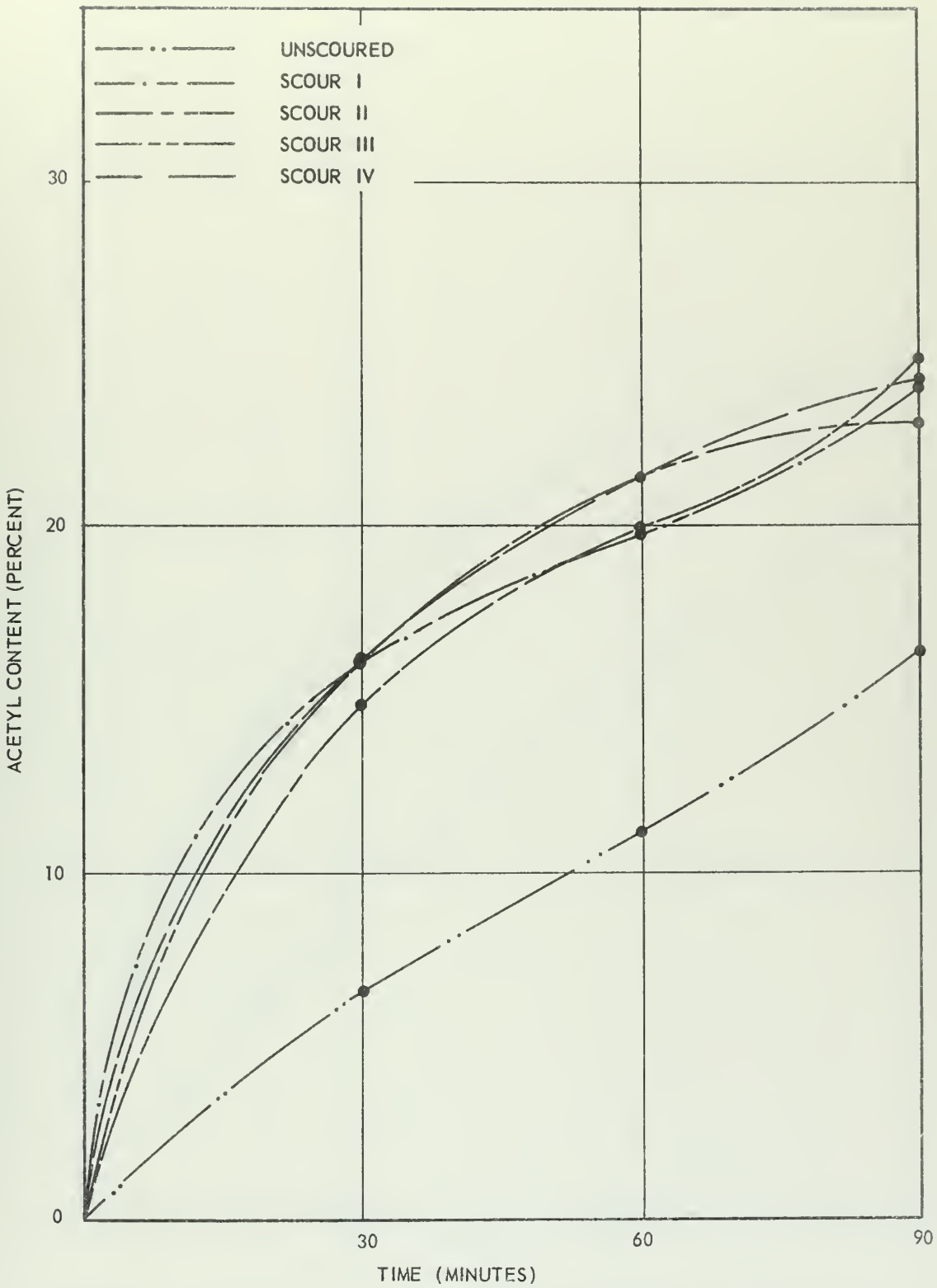

Figure 8. The Change of Acetyl Content With Time of Acetylation for Bob Shaw Cotton Unscoured and Scoured by Four Methods.

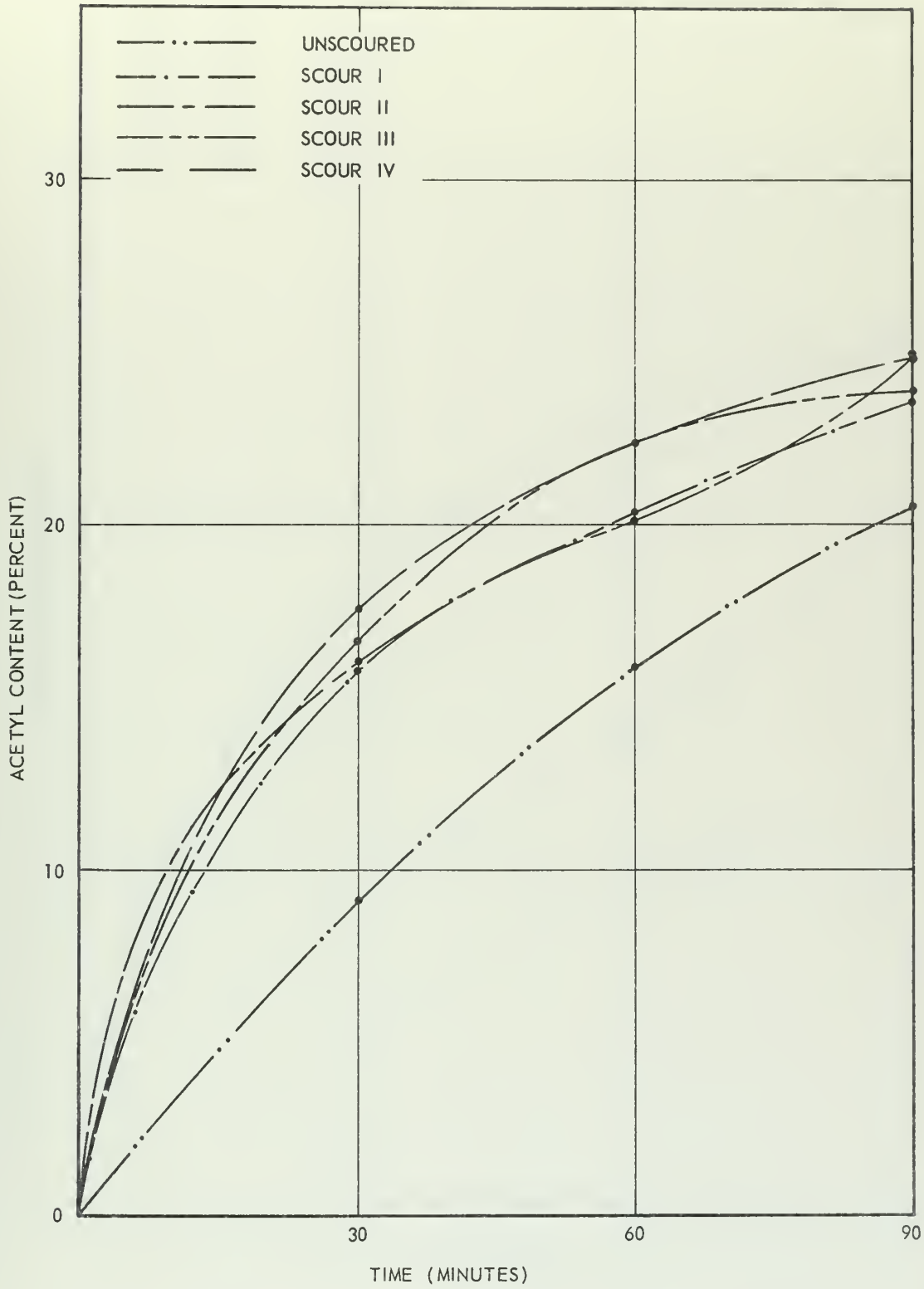

Figure 9. The Change of Acetyl Content With Time of Acetylation for Stoneville 2B Bale 249290 Cotton Unscoured and Scoured by Four Methods.

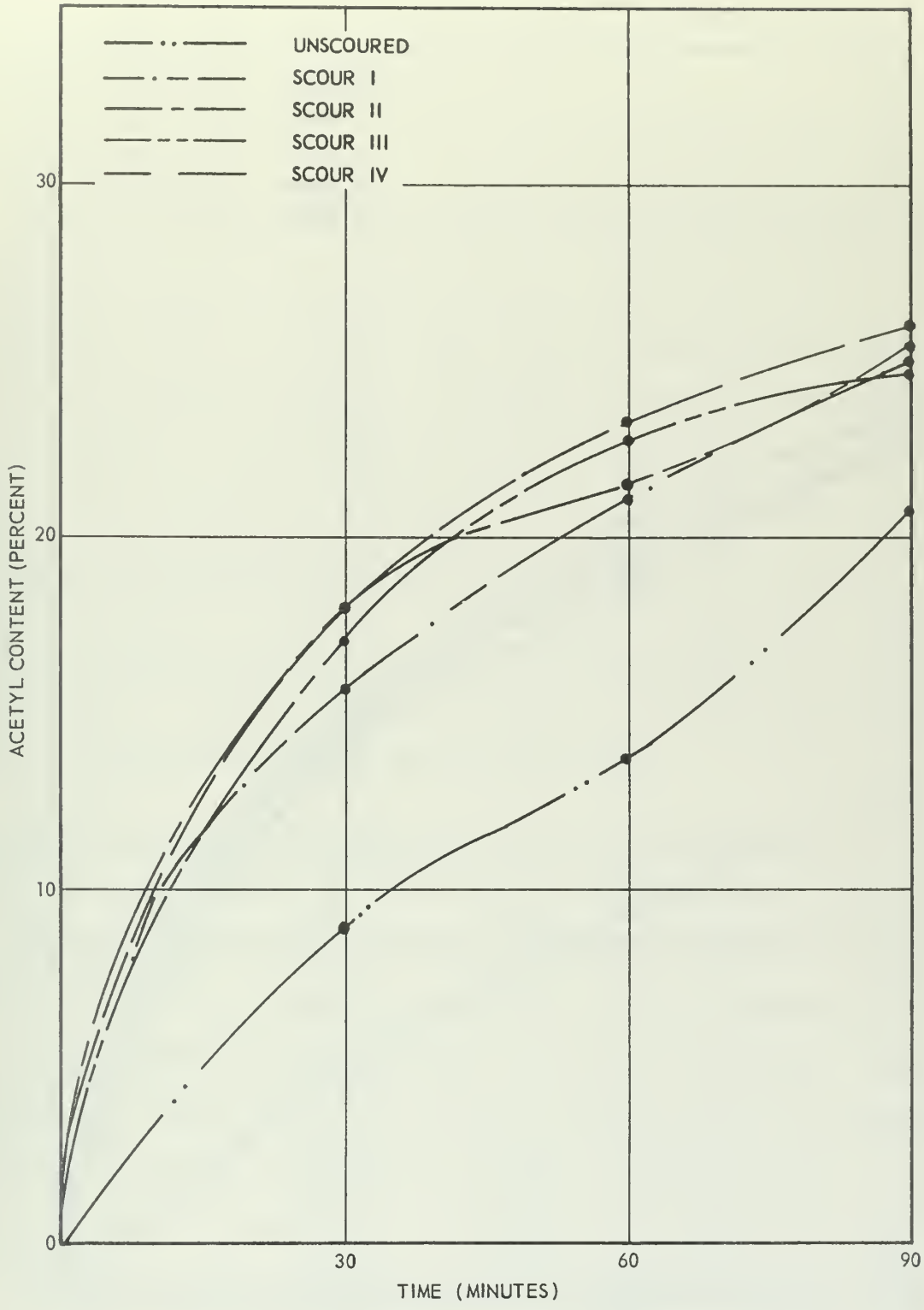

Figure 10. The Change of Acetyl Content With Time of Acetylation for Acala 1517 Cotton Unscoured and Scoured by Four Methods.

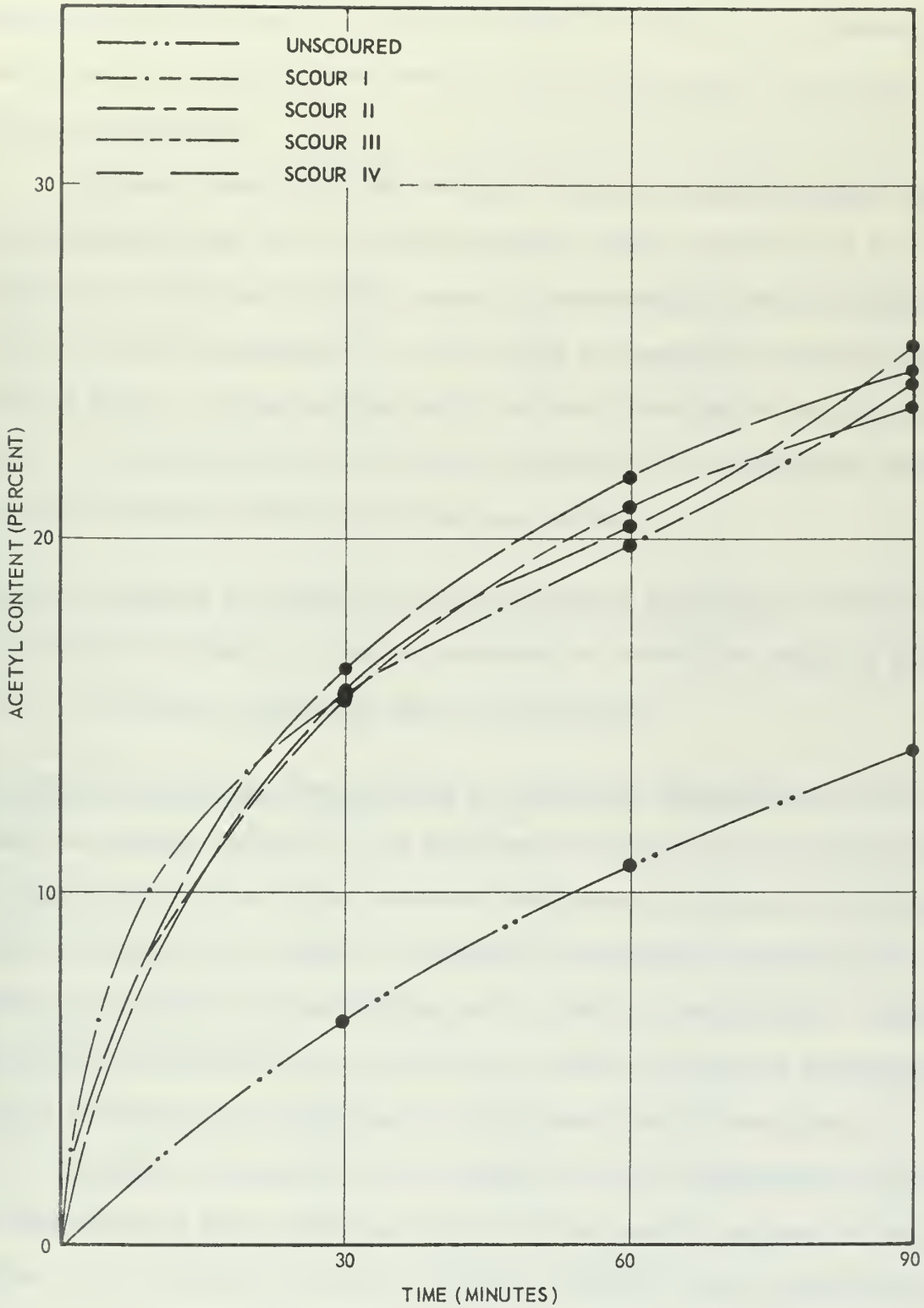

Figure 11. The Change of Acetyl Content With Time of Acetylation for Lockett 140 Cotton Unscoured and Scoured by Four Methods.

content of from 19.8 to 21.9 per cent in the same period of time, depending on the method of scouring employed. Thus, in this instance, the degree of acetylation was nearly doubled as a result of scouring before acetylation.

Although scour IV on the average produced a slightly higher degree of acetylation than the other three methods, there does not seem to be a significant difference in this respect. Considering a normal experimental error in the investigation, it is felt that all methods of scouring from mild to severe produced substantially the same increase in the rate and degree of acetylation and leveled the reactivity of the different varieties of cotton towards acetylation to the same extent.

Moisture Contents of Cottons at Various Relative Humidities.--The approximate moisture contents of the six varieties of cotton are shown in Table 5 for the humidity conditions under investigation.

The Effect of Moisture Conditioning on Subsequent Acetylation.--Table 5 shows the acetyl contents of the different varieties of cotton which were acetylated after first being moisture conditioned at different atmospheric relative humidities. Figures 12 through 17 illustrate graphically the effects of moisture preconditioning on the rate of acetylation. Figures 18 through 23 illustrate more clearly the effect of moisture preconditioning on the degree of acetylation for different times of reaction.

Although the results of this portion of the investigation are not as conclusive as those discussed above for the scoured cottons, it would appear that increasing moisture contents in the wide range corresponding

Table 5. Acetyl Contents of Cottons Partially Acetylated After Various Moisture Conditioning Pretreatments

Cottons	Moisture Content (%)	Acetylated at 64 F. Per cent acetyl after acetylation for: 30 minutes	60 minutes	90 minutes
15 PER CENT RELATIVE HUMIDITY:				
Memphis	4.1	12.3	18.4	20.7
Empire Bale 92	3.7	5.70	10.6	15.9
Bob Shaw	3.5	3.87	7.59	12.2
Stoneville 2B	4.2	6.59	11.7	16.8
Acala 1517	4.1	6.51	11.3	17.0
Lockett 140	3.4	3.40	6.84	11.7
35 PER CENT RELATIVE HUMIDITY:				
Memphis	4.2	12.2	17.6	20.9
Empire Bale 92	4.2	5.39	9.67	15.3
Bob Shaw	4.1	3.57	6.89	12.6
Stoneville 2B	4.3	6.54	10.7	17.1
Acala 1517	4.3	6.90	11.8	17.3
Lockett 140	3.9	3.51	6.87	11.4
50 PER CENT RELATIVE HUMIDITY:				
Memphis	5.0	12.6	19.4	24.3
Empire Bale 92	5.1	6.31	11.7	17.5
Bob Shaw	5.0	4.47	8.82	14.6
Stoneville 2B	5.3	7.64	13.0	19.2
Acala 1517	5.3	8.25	14.0	19.7
Lockett 140	5.2	3.90	8.51	13.0
65 PER CENT RELATIVE HUMIDITY:				
Memphis	5.8	14.0	19.5	23.5
Empire Bale 92	5.8	8.00	13.5	18.8
Bob Shaw	5.4	5.95	10.6	15.6
Stoneville 2B	5.9	9.37	15.1	19.2
Acala 1517	6.0	7.95	14.0	20.3
Lockett 140	5.9	6.32	10.8	13.5

Table 5. (Continued) Acetyl Contents of Cottons Partially Acetylated After Various Moisture Conditioning Pretreatments

Cottons	Moisture Content	Acetylated at 64 F. Per cent acetyl after acetylation for: 30 minutes	60 minutes	90 minutes
85 PER CENT RELATIVE HUMIDITY:				
Memphis	7.7	14.3	21.2	24.8
Empire Bale 92	8.1	8.26	14.7	18.6
Bob Shaw	7.5	5.76	11.3	15.6
Stoneville 2B	7.7	9.81	16.3	20.4
Acala 1517	8.4	11.4	19.1	22.9
Lockett 140	7.6	6.84	12.2	16.2

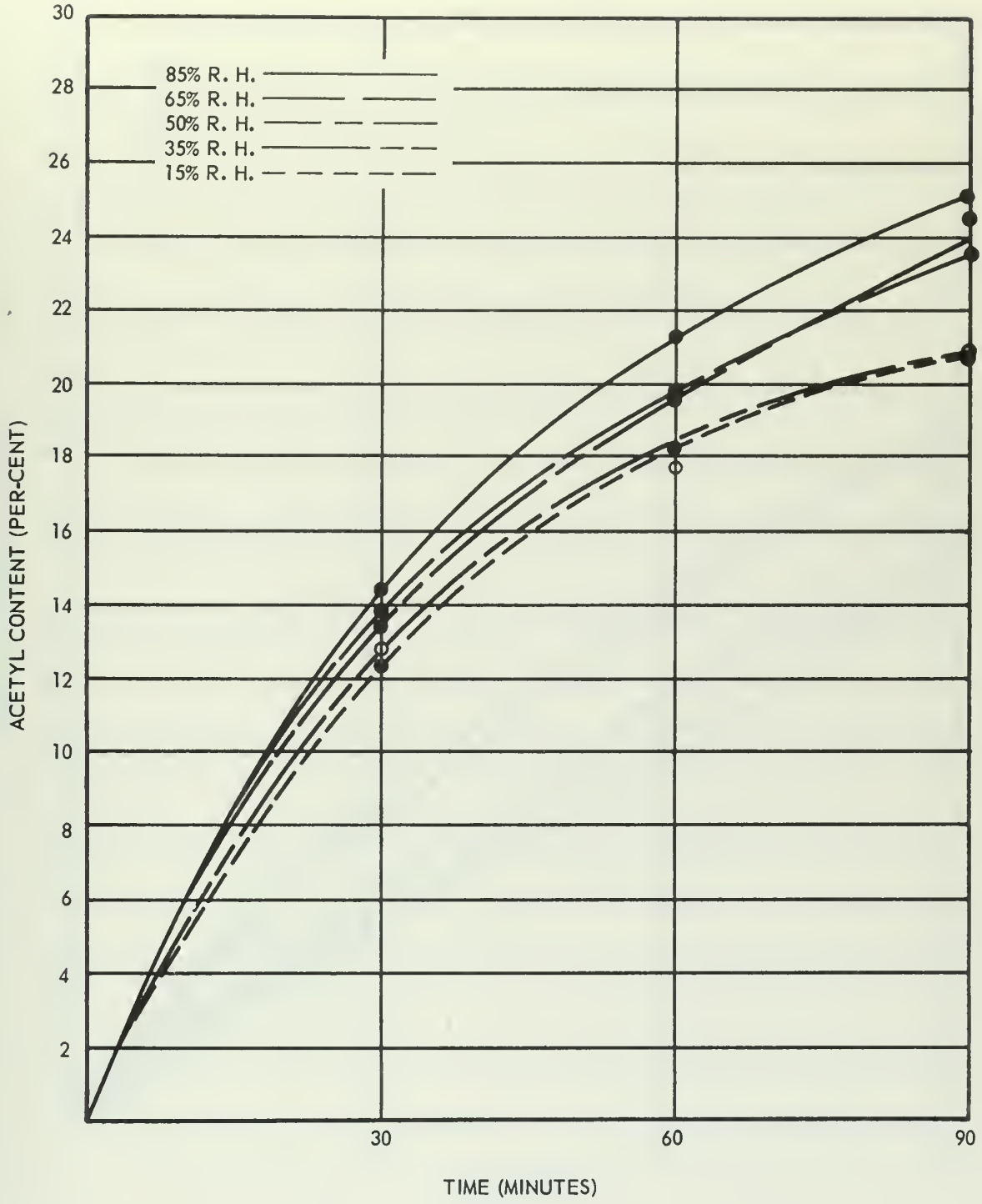

Figure 12. The Change in Acetyl Content with Time of Acetylation for Memphis Cotton Moisture Preconditioned at Five Different Relative Humidities.

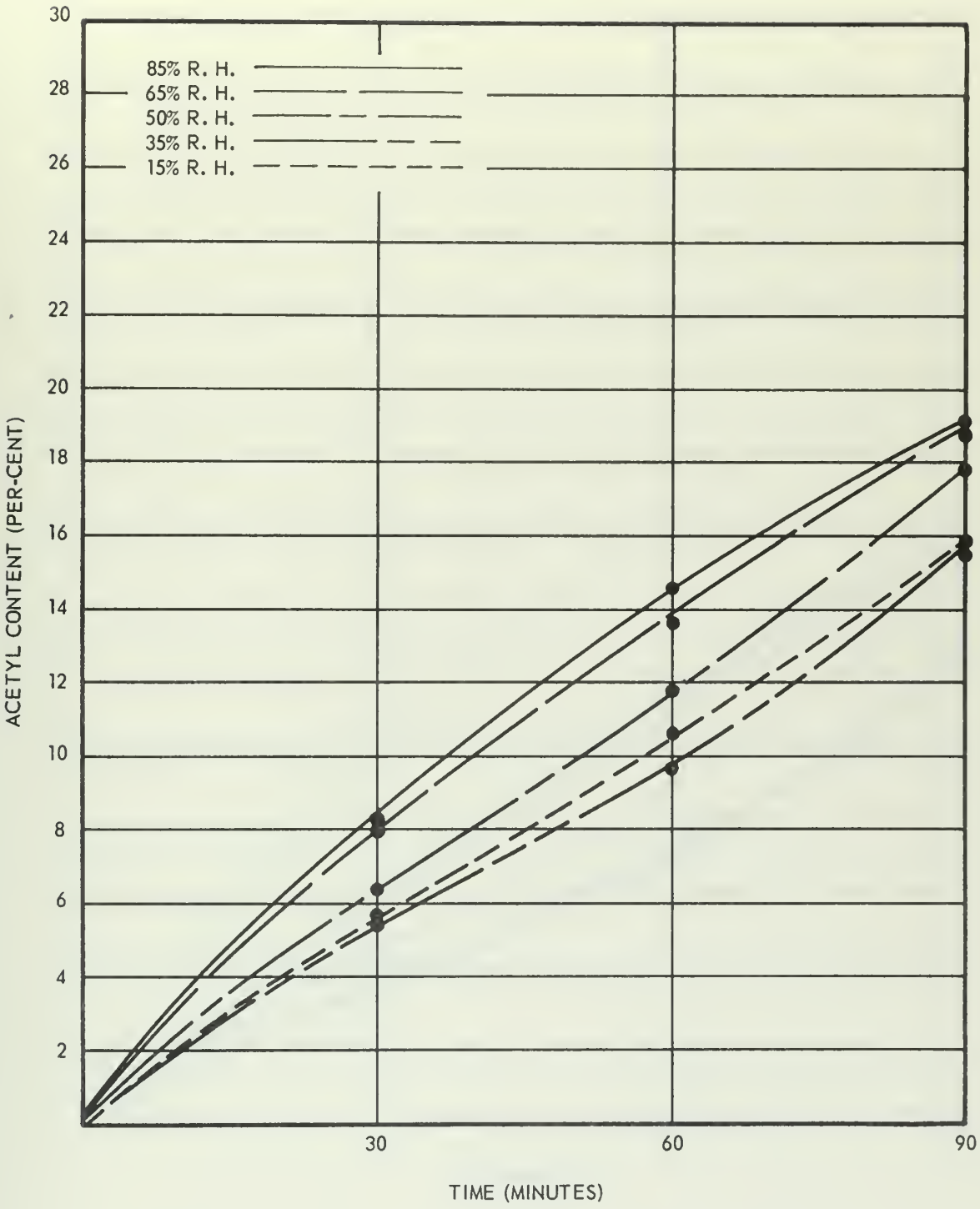

Figure 13. The Change in Acetyl Content with Time of Acetylation for Empire Bale 92 Cotton Moisture Preconditioned at Five Different Relative Humidities.

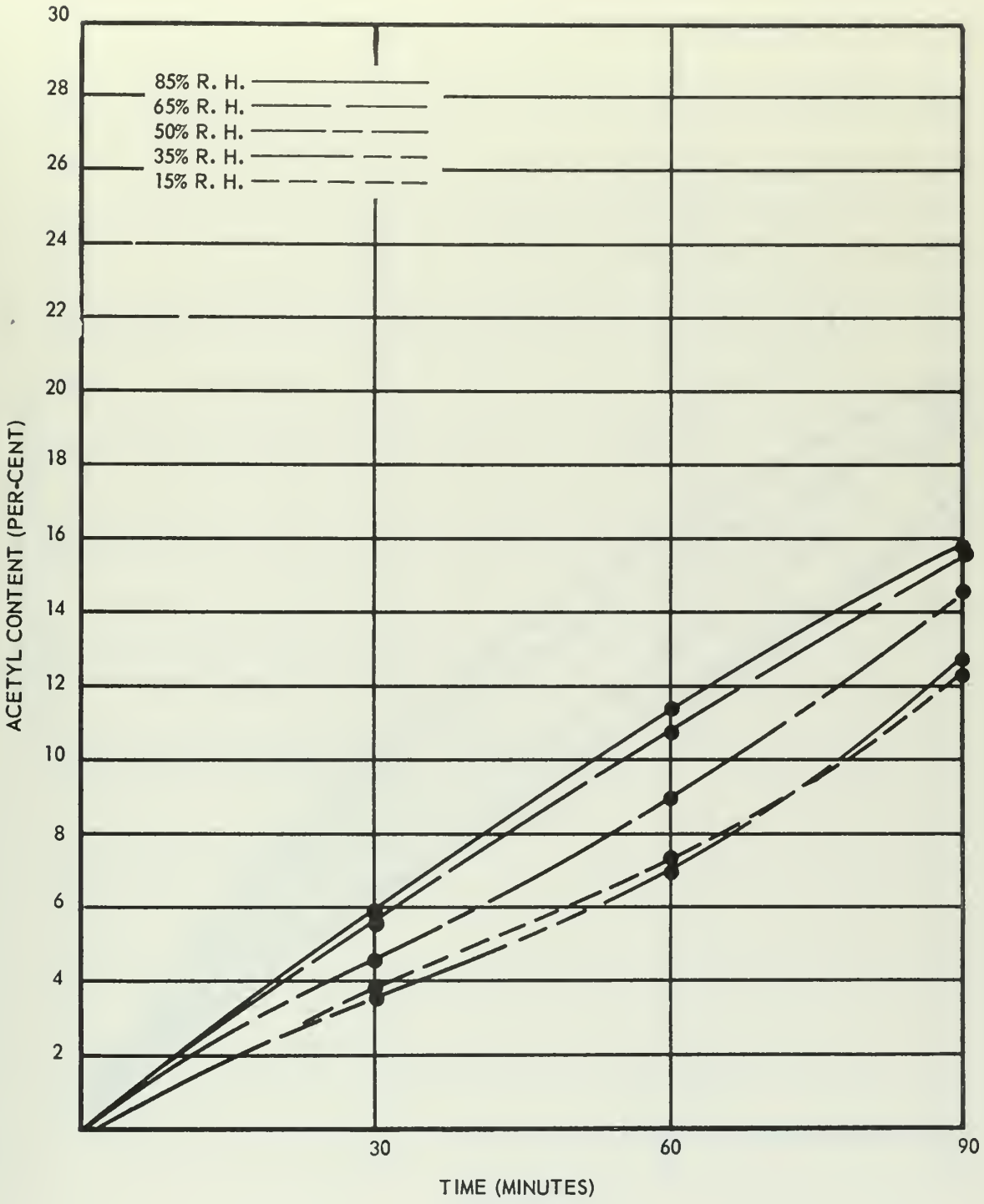

Figure 14. The Change in Acetyl Content with Time of Acetylation for Bob Shaw Cotton Moisture Preconditioned at Five Different Relative Humidities.

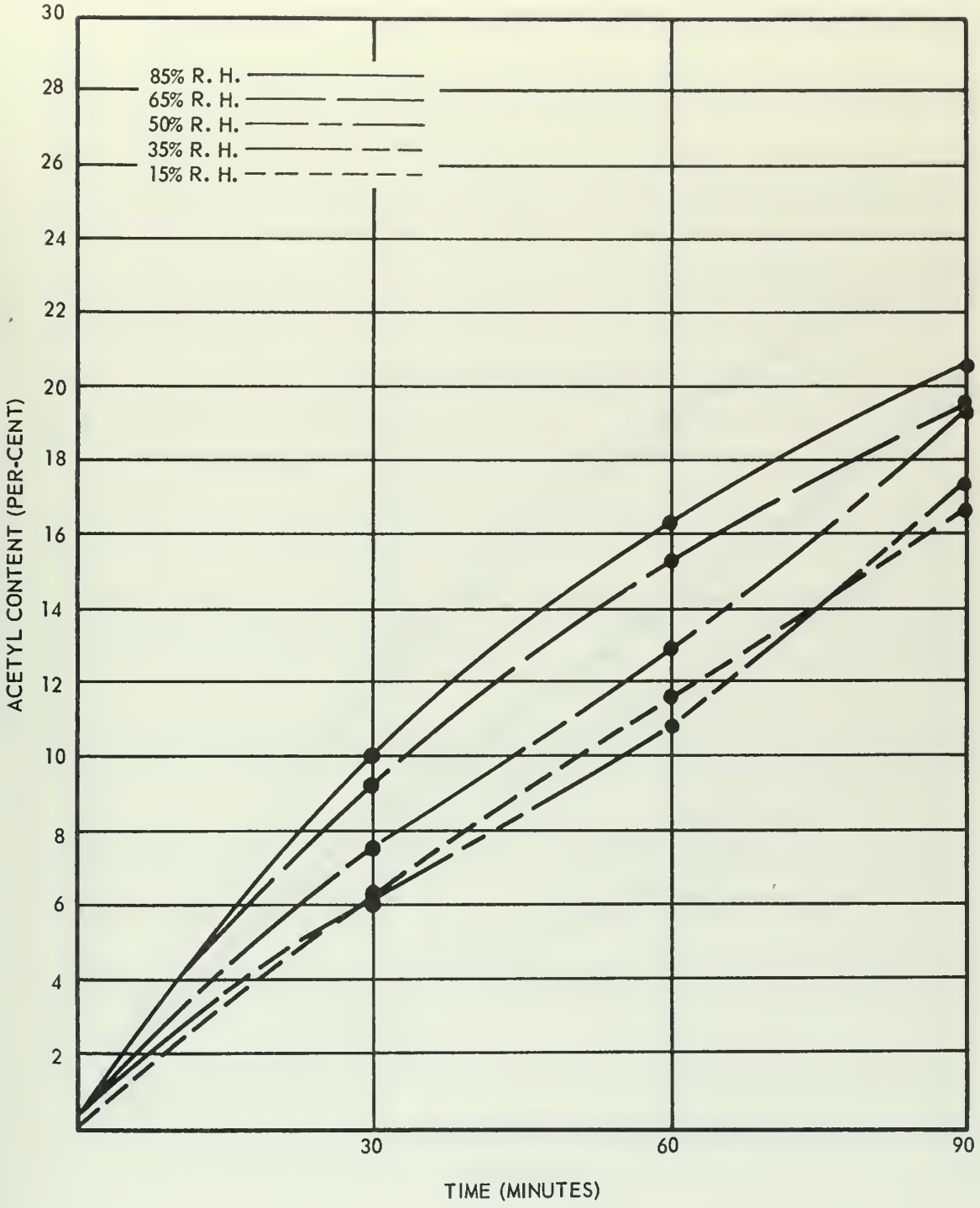

Figure 15. The Change in Acetyl Content with Time of Acetylation for Stoneville 2B Cotton Moisture Preconditioned at Five Different Relative Humidities.

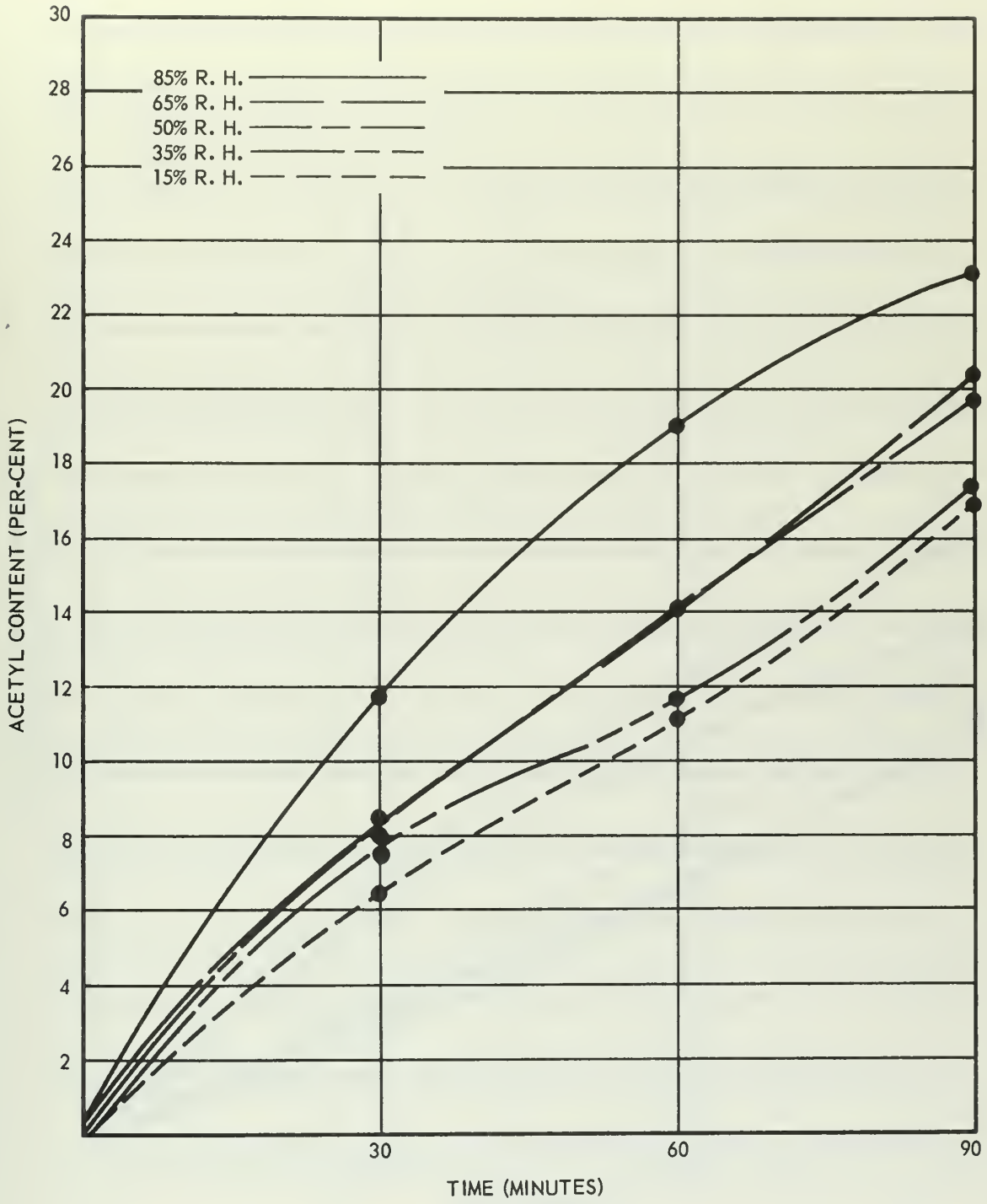

Figure 16. The Change in Acetyl Content with Time of Acetylation for Acala 1517 Cotton Moisture Preconditioned at Five Different Relative Humidities.

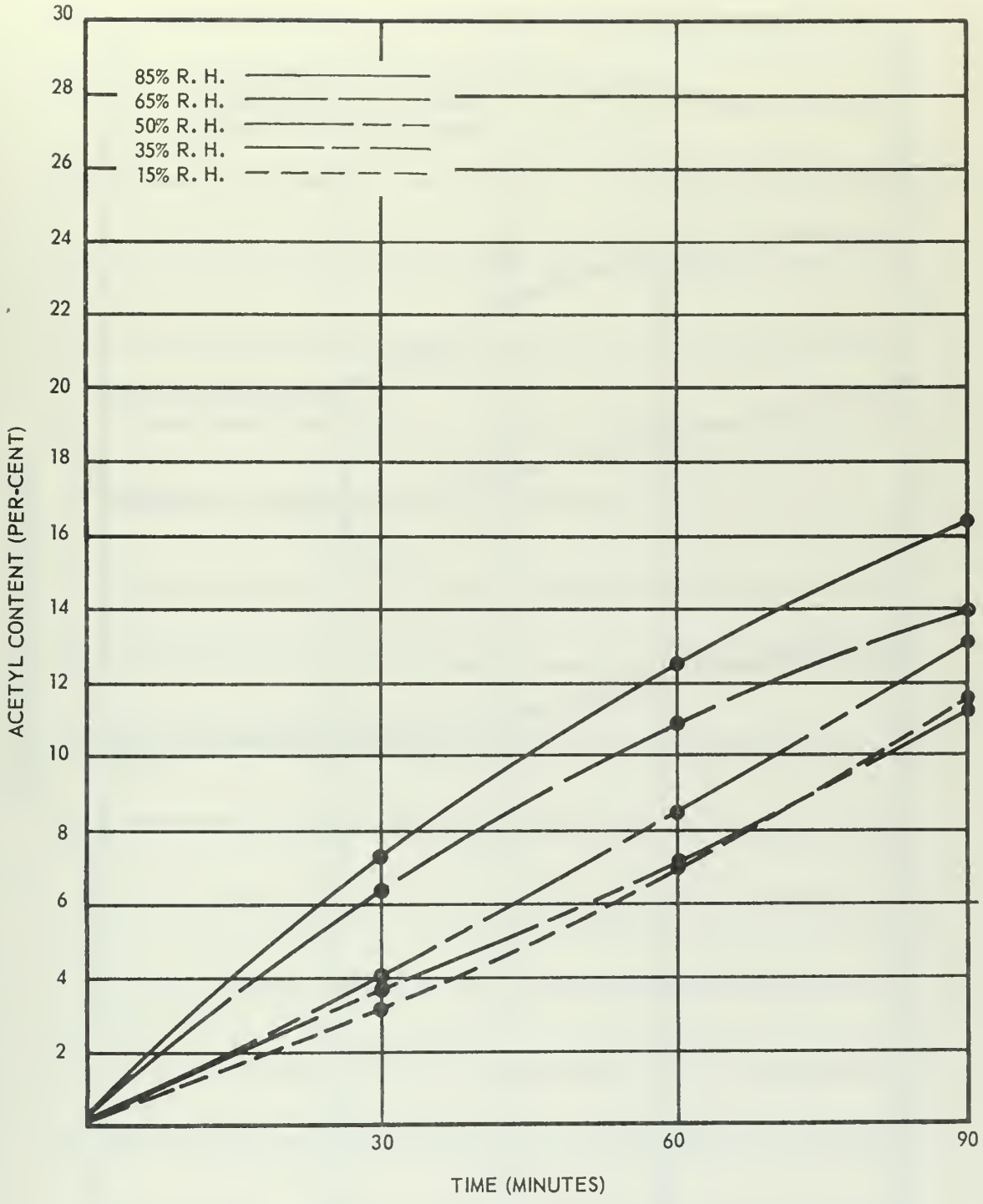

Figure 17. The Change in Acetyl Content with Time of Acetylation for Lockett 140 Cotton Moisture Preconditioned at Five Different Relative Humidities.

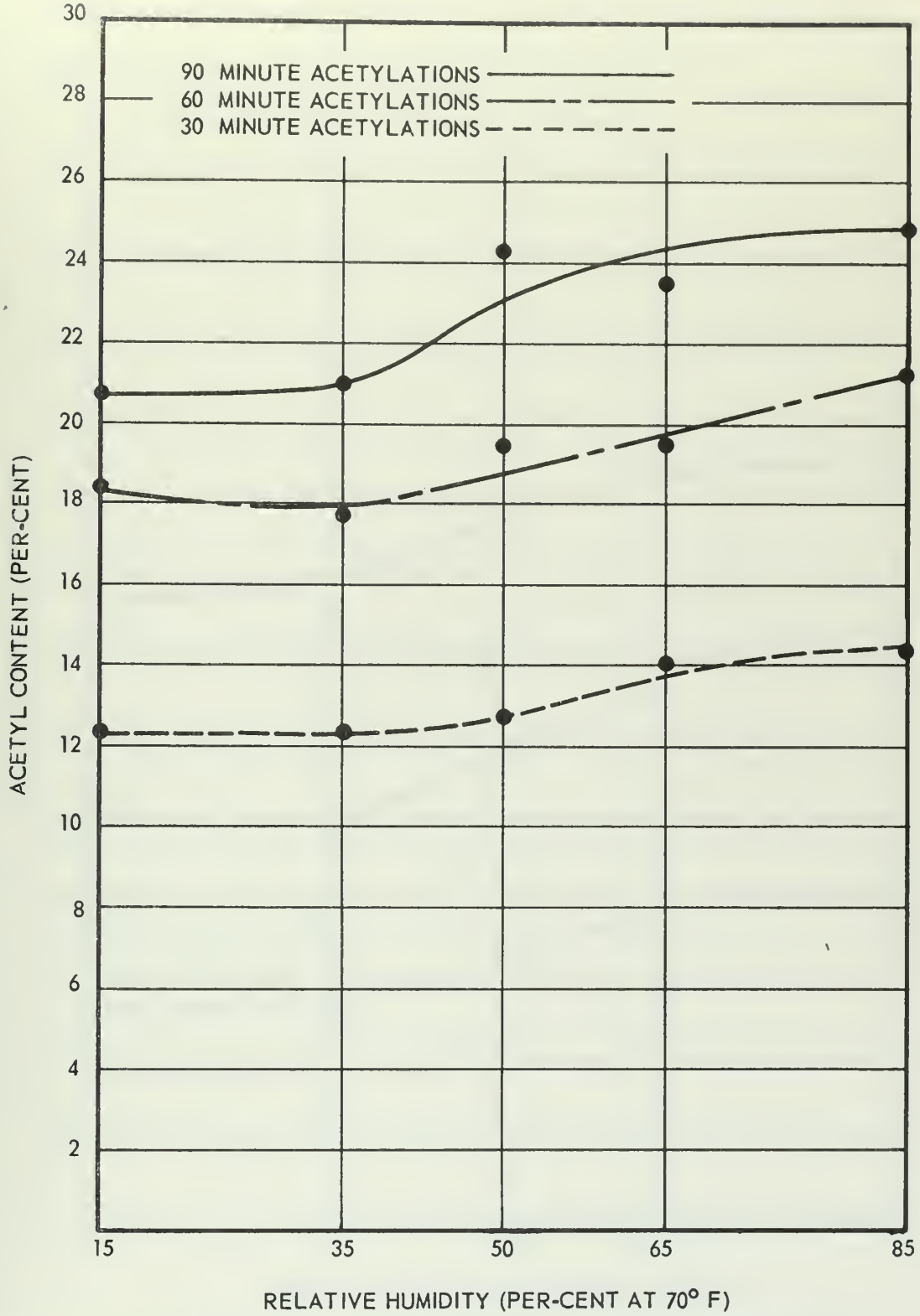

Figure 18. The Change in Acetyl Content with Relative Humidity of Preconditioning for Memphis Cotton at Three Times of Acetylation.

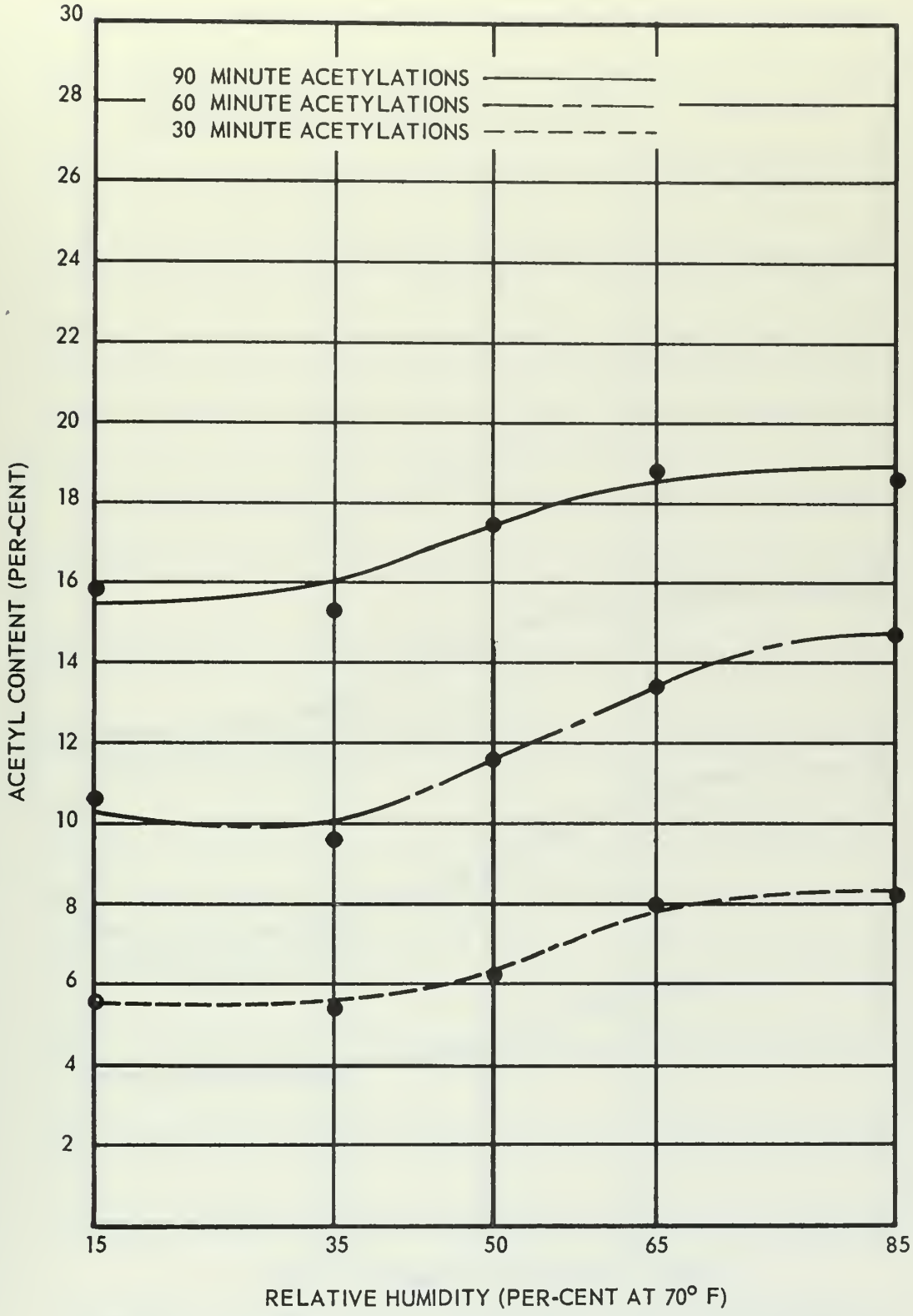

Figure 19. The Change in Acetyl Content With Relative Humidity of Preconditioning for Empire Bale 92 Cotton at Three Times of Acetylation.

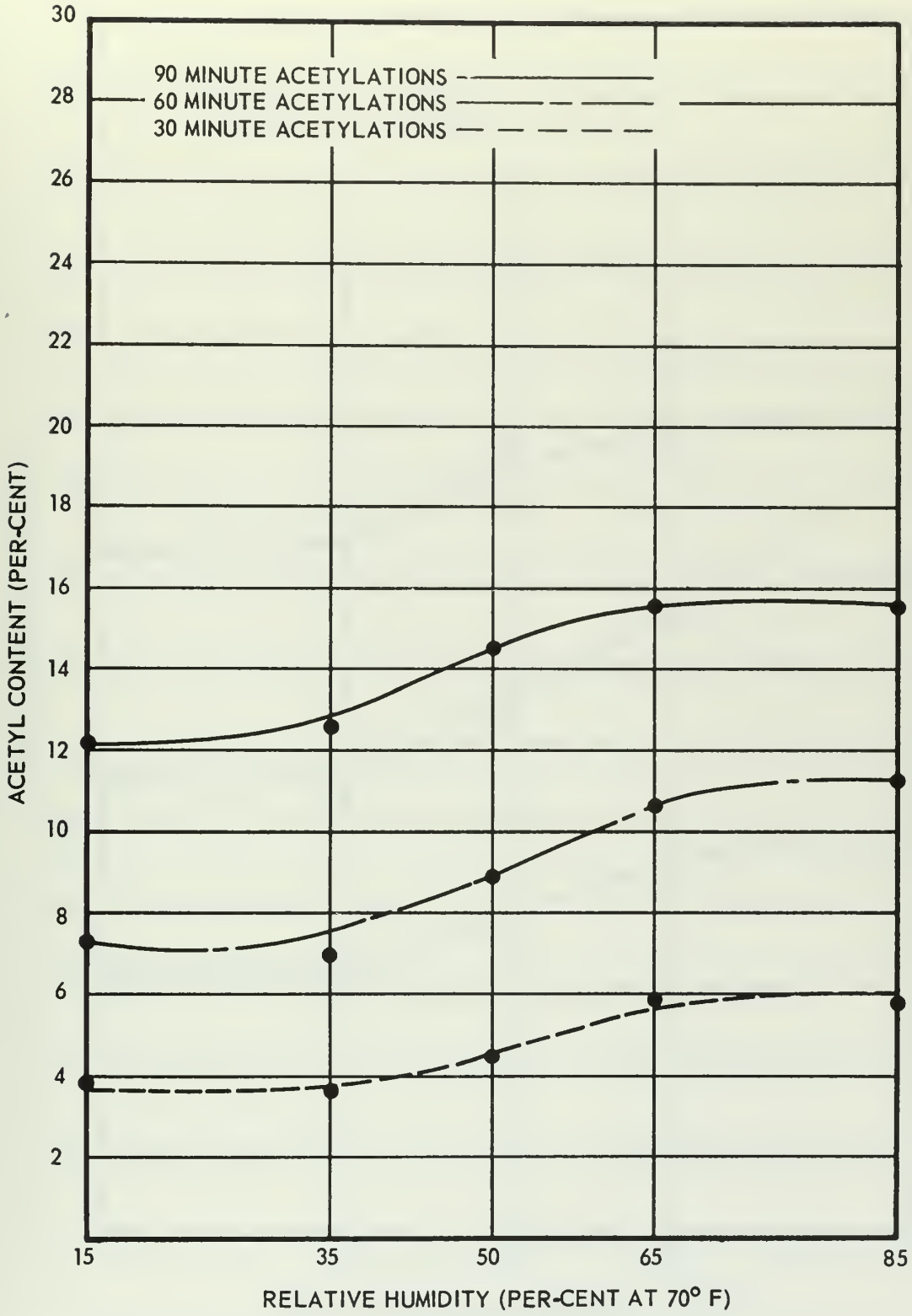

Figure 20. The Change in Acetyl Content with Relative Humidity of Preconditioning for Bob Shaw Cotton at Three Times of Acetylation.

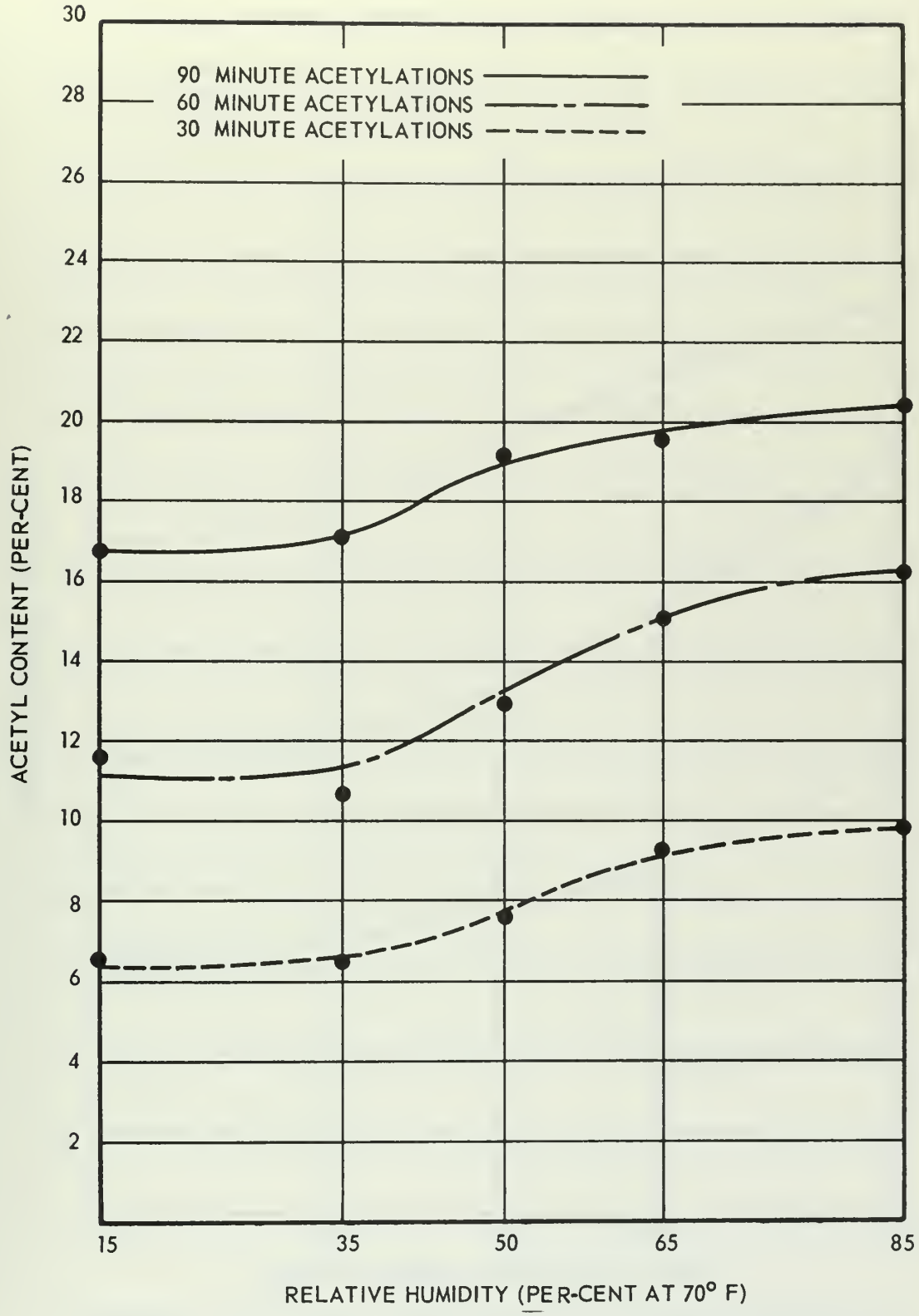

Figure 21. The Change in Acetyl Content with Relative Humidity of Preconditioning for Stoneville 2B at Three Times of Acetylation.

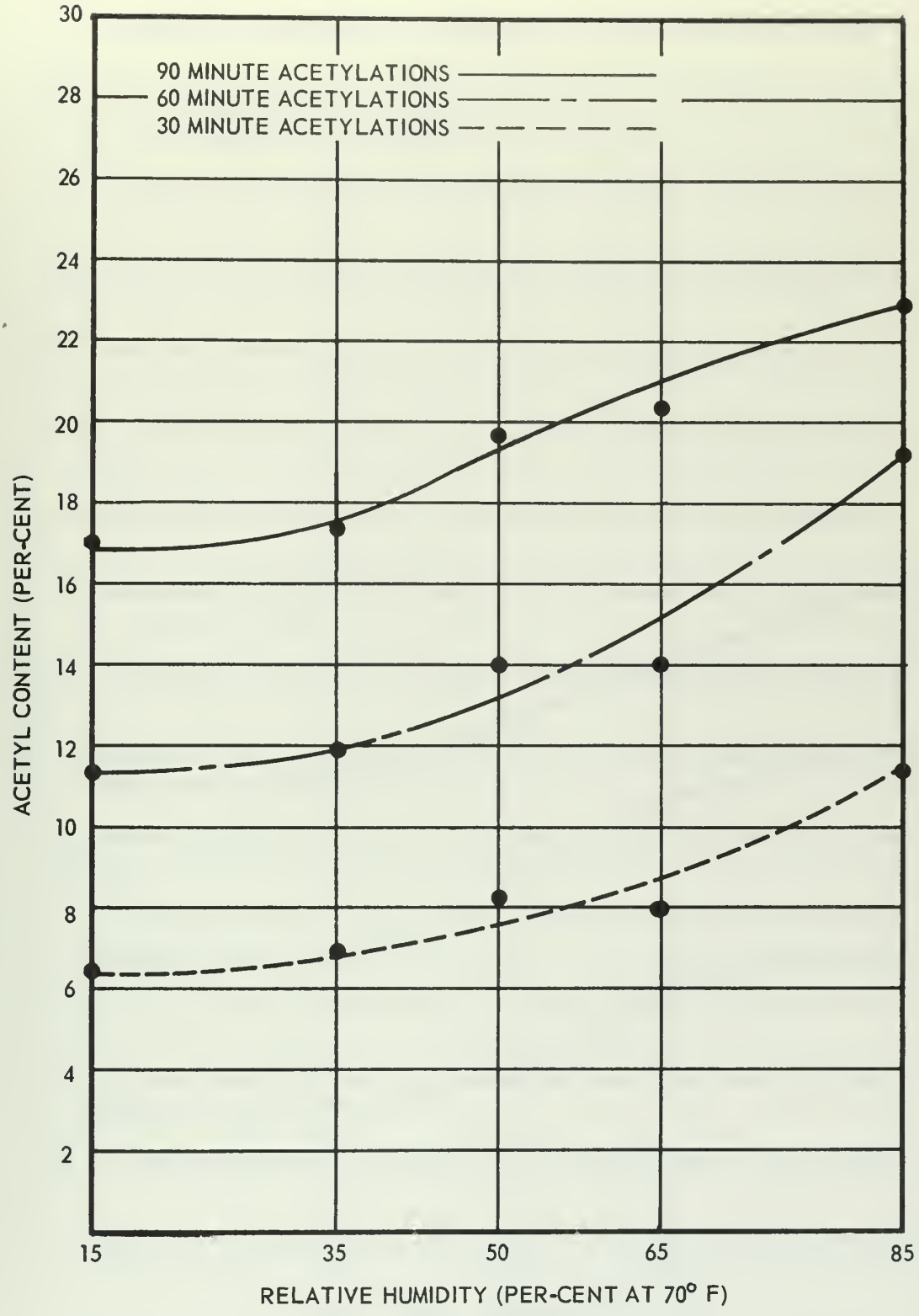

Figure 22. The Change in Acetyl Content with Relative Humidity of Preconditioning for Acala 1517 Cotton at Three Times of Acetylation.

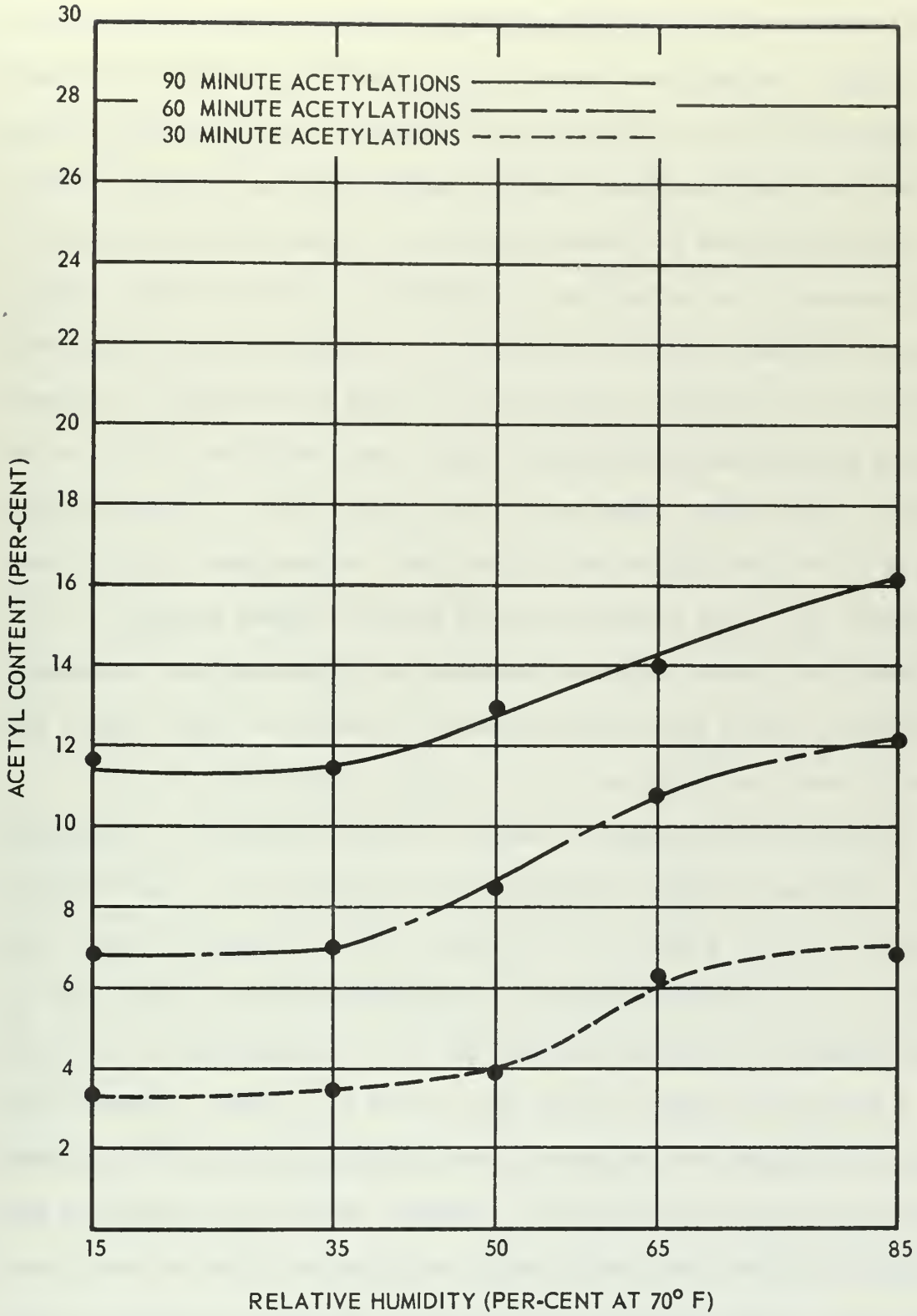

Figure 23. The Change in Acetyl Content with Relative Humidity of Preconditioning for Lockett 140 Cotton at Three Times of Acetylation.

to 35 to 85 per cent relative humidity at 70° F. increase the rate and degree of acetylation for all cottons under investigation. Again the mature cottons are more affected by pretreatment than the relatively immature Memphis variety. Taking the same example as used previously in discussing the results of scouring, Lockett 140 cotton exhibits a steady, almost linear rise in degree of acetylation at 60 minutes with increasing moisture content. At 35 per cent relative humidity an acetyl content of only 6.87 per cent is reached after acetylating for 60 minutes, while 50, 65, and 85 per cent relative humidity preconditioning produce acetyl contents of 8.51, 10.8, and 12.2 per cent, respectively, for the same period of acetylation. The rise is less regular and not as pronounced for the immature Memphis variety and the irrigated Acala 1517 variety, suggesting that immaturity and moisture conditions during the growth of the cotton alter the effect of moisture conditioning prior to acetylation.

For all varieties of cotton it is to be noted that there is no appreciable difference in rate or degree of acetylation produced by preconditioning at 15 and 35 per cent atmospheric relative humidity. This somewhat unexpected result might be explained by assuming that the lower degree of swelling of cottons conditioned at relative humidities of 35 per cent and below is compensated for by the swelling effect of the glacial acetic acid presoak. That is, it may be that glacial acetic acid exerts a swelling effect on cotton approximately equal to that produced by conditioning at 35 per cent relative humidity. If this is the case, then cottons conditioned at relative humidities below 35 per cent would be brought to the same degree of swelling by the action of glacial acetic acid presoaking, whereas cottons conditioned at relative humidities above 35 per cent

would retain in glacial acetic acid the increased degree of swelling produced by their increased moisture contents.

The Effect of Physical Properties of the Cottons on Acetylation.--The physical properties of the cottons selected for experimentation are shown in Table 2. An examination of this table reveals that a wide range in maturity values, fiber fineness and other properties are demonstrated in the fibers selected. From an analysis of acetyl contents of the different varieties of cotton as presented in Tables 4 and 5 it is evident that a definite relationship exists between maturity, fiber fineness, and rate of acetylation. The acetyl content of the immature Memphis cotton is consistently higher than any other cottons acetylated under similar circumstances in this study. The two most mature cottons, Bob Shaw and Lockett 140, appear to be the least responsive to acetylation treatments. This is particularly true for the unscoured samples. It should be noted that the Micronaire readings of Bob Shaw and Lockett 140 are the highest of this group of cottons. Although the maturity of the three remaining cottons range from 72 to 86 per cent, very little difference can be observed in their reaction toward acetylation treatments. Results of the differential dyeing test indicated that all cottons were evenly acetylated.

Changes in Physical Properties Due to Acetylation.--Tables 6 through 17 in the Appendix present the analytical data on fiber fineness, fiber strength, and moisture content at standard condition for all cottons acetylated in the course of this study. These results were furnished

by Mr. Benjamin Gunby Holloway. For convenient reference, the acetyl contents of the acetylated cottons are also given in these tables. For a detailed analysis of the effects of acetylation on the physical properties of cotton the reader is referred to the unpublished Masters Thesis presented by Mr. Holloway.[24]

In general, it may be stated that with increasing acetyl contents the cottons under investigation exhibited a slightly decreased tensile strength, a marked decrease in moisture content at standard condition, and a slight swelling or increase in fiber diameter as measured by the Sheffield Micronaire.

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[24]B. G. Holloway, A Comparison of the Physical Characteristics of Partially Acetylated Cottons with Those of Untreated Cottons, Masters Thesis, Georgia Institute of Technology, 1953.

CHAPTER VI

CONCLUSIONS

Scouring before acetylation by any one of the methods employed in this investigation tends to minimize the differences in the rate and degree of acetylation of different varieties of cotton.

Scouring before acetylation by any one of the methods employed in this investigation increases the rate and degree of acetylation of all varieties of cotton, and especially increases the reactivity of the more mature varieties.

Within the range of severity of this investigation, a mild scour using no caustic produces substantially the same increase in rate and degree of acetylation and levels the reactivity of the different varieties of cotton towards acetylation to the same extent as a severe caustic scour.

All varieties of cotton investigated, and particularly those with a high maturity, displayed an increase in the rate and degree of acetylation with increased moisture contents when conditioned before acetylation to moisture contents corresponding to a range of atmospheric conditions from 35 to 85 per cent relative humidity at 70° F.

For the varieties of cotton investigated, there was no appreciable difference in the rate or degree of acetylation produced by conditioning before acetylation to moisture contents corresponding to 15 and 35 per cent atmospheric relative humidity at 70° F.

CHAPTER VII

RECOMMENDATIONS FOR FURTHER STUDY

It is recommended that the effect of additional pretreatments on the rate and degree of acetylation of cotton be investigated. Any pretreatment, such as mercerizing, which results in a swelling of the cotton fiber should increase the rate and degree of acetylation. In addition, purification treatments such as bleaching should also have a beneficial effect on the rate of subsequent acetylation.

Further study in the field of moisture conditioning and presoaking should be undertaken based on the results of this and companion investigations.[25] The effects of extremely low as well as extremely high moisture contents should be more accurately determined. An investigation of the effects of moisture contents approaching those corresponding to 100 per cent relative humidity should be particularly fruitful. In addition, the effect of presoaking in an acetic acid-water mixture should be investigated. It may be, for example, that presoaking in a 90 per cent acetic acid - ten per cent water solution would produce an increase in the rate of acetylation without regard to the previous moisture history of the cottons employed.

As sufficient data on individual pretreatments accumulates, the various treatments should be investigated in combination. If the beneficial

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[25] E. D. Owens, A Study of the Effect of Time and Temperature of Presoaking and the Temperature of Acetylation on the Rate and Degree of Partial Acetylation of Different Varieties of Cotton Fibers, Masters Thesis, Georgia Institute of Technology, 1953.

effects of various individual pretreatments is cumulative, it may be that the reaction of partial acetylation could be applied successfully to a continuous range fabric operation, rather than to the less economical batch process. In the light of present knowledge, such a range should include a scouring operation, a water dip - squeeze, a hot (130-170 F.) glacial acetic acid presoak, and a low (60-70 F.) temperature acetylation bath.

The results of this study should be confirmed employing yarns and fabrics rather than raw stock.

BIBLIOGRAPHY

A.S.T.M. Committee D-13, A.S.T.M. Standards on Textile Materials. Philadelphia: American Society for Testing Materials, 1950. 558 pp.

Blow, C. M. and Knight, W. J., "Textile Fibres and Rubber as Associated Materials for Manufacture," Journal of the Textile Institute, 35:12, 1944.

Callow, H. J., "Acetylation of Jute," Journal of the Textile Institute, 43: T423-432, 1952.

Chippindale, E., "Low Acetylation of Cellulose Fibers," Journal of the Society of Dyers and Colurists, L: 142-149, 1934.

Cooper, A. S., Voorhies, S. T., Buras, E. M., and Goldthwait, C. F., "Cotton: Partial Acetylation," Textile Industries, 116: 97-102, 1952.

Dean, J. D., Strickland, W. B., and Berard, W. N., "The Soil Burial Test, S.R.R.L., New Orleans," American Dyestuff Reporter, 34: 195-201, 1945.

Elod, E., Schmid-Bielenberg, H., and Thoria, L., "Cellulose Fibers: Acetylation," Agnew Chem., 47: 465-468, 1934, through Journal of the Textile Institute, 25: A420, 1934.

Goldthwait, C. F., McLaren, J., and Voorhies, S. T., "Acetylated Cotton Highly Resistant to Rotting," Textile World, 96: 115-117, 1946.

Hermans, P. H., Physics and Chemistry of Cellulose, New York: Elsevier Publishing Co., Inc., 1949. 534 pp.

Hermans, P. H. and A. Weidinger, "X-Ray Studies on the Crystallinity of Cellulose," Journal of Polymer Science, 4: 135-144, 1949.

Heuser, Emil, The Chemistry of Cellulose. New York: John Wiley and Sons, Inc., 1944. 660pp.

Heuser, E., "Cellulose: Acetylation in Phosphoric Acid Solution," Industrial and Engineering Chemistry, 40: 1500-1506, 1948.

Heuser, E., "Factors Which Influence the Kinetics of Cellulose Reactions," Textile Research Journal, 20: 829-836, 1950.

Honold, E., J. Poynot, and A. Cuculla, "Heat Resistance of Partially Acetylated Cotton Fabrics," Textile Research Journal, 22: 25-29, 1952.

Howlett, F. and E. Martin, "Acetylated Celluloses: Acetic Acid Yield; Determination," Journal of the Textile Institute, 35: T1-6, 1944.

Hvattum, O. J., and H. A. Turner, "Grey Cotton: Removal of Impurities," Journal of the Society of Dyers and Colurists, 67: 416-424, 1951.

Kanamaru, K., "Cellulose Acetate and Nitrocellulose: Refractive Index," Helvetica Chim. Acta., 17: 1429-1440, 1934, through Journal of the Textile Institute, 26: A222, 1935.

Knight, H. G., "Cotton - Yesterday, Today and Tomorrow," Chemical Engineering News, 20: 581-583, 1942.

Mackenzie, H. A. E., "Acetic Acid. Acetic Anhydride System: Properties," Transactions of the Faraday Society, 44: 159-171, 1948.

Malm, C. J., K. T. Barkey, D. C. May, and E. B. Lefferts, "Treatment of Cellulose Prior to Acetylation," Industrial and Engineering Chemistry, 44: 2904-2909, 1952.

Malm, C. J. and H. T. Clark, "The Action of Fatty Acids on Cellulose," Journal of the American Chemical Society, 51: 274, 1929.

Mark, H., "Intermicellar Hole and Tube System in Fiber Structure," Journal of Physical Chemistry, 44: 779, 1940.

Marsh, J. T., An Introduction to Textile Bleaching, New York: John Wiley and Sons, Inc., 1948. 512 pp.

Marsh, J. T. and F. C. Wood, An Introduction to the Chemistry of Cellulose. London: Chapman and Hall, Ltd., 1938. 431 pp.

Matthews, J. M., Textile Fibers. New York: John Wiley and Sons, Inc., 1947. 1133 pp.

Meller, A., "Fibrous Cellulose: Reactivity," Journal of Polymer Science, 4: 619-628, 1949.

Meyer, K. H., Natural and Synthetic High Polymers. New York: Interscience Publishers, Inc., 1950. 393 pp.

Morehead, F. F., "A Method for Studying the Effect of Humidity on the Cross-sectional Swelling of Some Common Fibers," Textile Research Journal, 22: 535-539, 1952.

O'Brien, F. E. M., "Humidity Control Solutions," Journal of Scientific Instruments, 25: 73-76, 1948.

Ott, Emil, Cellulose and Cellulose Derivatives. New York: Interscience Publishers, Inc., 1946. 1176 pp.

Petitpas, G., "The Perchloric Acid Catalysis of the Acetylation of Cellulose," Mem. services chim. etat., 31: 178-186, 1944. through Chemistry Abstracts, 40: 7613, 1946.

Pettinger, A. H., "The Metallic Perchlorates," American Dyestuff Reporter, 27: 122-136, 1938.

Plunguian, M., Cellulose Chemistry. Brooklyn: Chemical Publishing Co., Inc., 1943. 97 pp.

Poon, S. Y., "A Study of the Rate and Degree of Acetylation of Different Varieties of Cotton Fibers." Masters Thesis, Georgia Institute of Technology, 1952.

Rollins, M. L., H. Fort, and D. Skau, "Cellulose - Water Relations," U. S. Department of Agriculture, Library List No. 44, 1949. 63 pp.

Scott, W. M., "Some Recent Developments in Cotton Research at S.R.R.L.," Textile Research Journal, 19: 436-443, 1949.

Sheffield Corporation, "Instructions for the Use of the Sheffield Micronaire," Dayton, Ohio: Sheffield Corporation.

Sisson, W. A., "X-Ray Diffraction Behavior of Cellulose Derivatives," Industrial and Engineering Chemistry, 30: 530-532, 1938.

Skinkle, J. H., Textile Testing. Brooklyn: Chemical Publishing Co., 1949. 353 pp.

Stokes, R. H. and R. A. Robinson, "Standard Solutions for Humidity Control at 25 C.," Industrial and Engineering Chemistry, 41: 2013, 1949.

Ward, K., "Crystallinity of Cellulose and its Significance for the Fiber Properties," Textile Research Journal, 20: 363-372, 1950.

Wright, R. E. and M. Harris, "Textile Industry Changes Pace," Chemical Engineering News, 28: 38-39, 1950.

APPENDIX

Table 6. Physical Data for Memphis Cotton Partially Acetylated After Different Methods of Scouring

Time of Acetylation	Per cent Acetyl	Fiber Fineness (Micronaire)	Strength (Pressley)	Moisture Content (%)
SCOUR I:				
Unacetylated	-	2.50	6.98	4.8
30 minutes	18.3	2.70	6.73	2.5
60 minutes	20.8	2.91	6.28	3.0
90 minutes	26.1	3.13	5.86	2.3
SCOUR II:				
Unacetylated	-	2.50	7.07	4.9
30 minutes	17.9	2.62	6.42	2.2
60 minutes	21.2	2.73	6.22	2.7
90 minutes	26.1	2.98	5.38	1.9
SCOUR III:				
Unacetylated	-	2.50	6.98	5.6
30 minutes	19.2	2.68	6.17	3.7
60 minutes	23.2	2.75	5.45	3.8
90 minutes	24.6	2.95	5.33	2.2
SCOUR IV:				
Unacetylated	-	2.53	7.04	5.2
30 minutes	18.3	2.63	6.00	4.2
60 minutes	23.7	2.92	5.78	3.6
90 minutes	25.4	2.95	5.44	2.6

Table 7. Physical Data for Empire Bale 92 Cotton Partially Acetylated After Different Methods of Scouring

Time of Acetylation	Acetyl (%)	Fiber Fineness (Micronaire)	Fiber Strength (Pressley)	Moisture Content
SCOUR I:				
Unacetylated	-	3.71	6.80	4.6
30 minutes	16.3	4.03	6.41	2.5
60 minutes	20.9	4.32	5.70	3.0
90 minutes	24.2	4.40	5.58	1.7
SCOUR II:				
Unacetylated	-	3.65	6.89	4.6
30 minutes	16.4	3.77	6.43	2.5
60 minutes	20.6	4.13	5.97	2.6
90 minutes	25.3	4.20	5.40	1.6
SCOUR III:				
Unacetylated	-	3.72	7.04	5.0
30 minutes	17.0	3.98	6.15	3.6
60 minutes	22.0	4.10	5.72	3.4
90 minutes	23.7	4.40	5.52	2.6
SCOUR IV:				
Unacetylated	-	3.70	6.95	5.0
30 minutes	16.7	4.00	6.48	4.0
60 minutes	22.9	4.02	6.15	3.5
90 minutes	24.5	4.20	5.99	2.8

Table 8. Physical Data for Bob Shaw Cotton Partially Acetylated After Different Methods of Scouring

Time of Acetylation	Acetyl (%)	Fiber Fineness (Micronaire)	Fiber Strength (Pressley)	Moisture Content (%)
SCOUR I:				
Unacetylated	-	5.07	8.22	4.9
30 minutes	16.1	5.13	6.69	2.6
60 minutes	19.7	5.67	6.45	2.8
90 minutes	23.8	5.80	5.61	1.7
SCOUR II:				
Unacetylated	-	5.00	8.07	4.4
30 minutes	14.9	5.02	6.43	2.2
60 minutes	20.0	5.55	6.39	3.0
90 minutes	24.8	5.50	5.87	1.7
SCOUR III:				
Unacetylated	-	5.03	7.84	4.8
30 minutes	16.0	5.35	6.53	3.3
60 minutes	21.5	5.52	6.40	3.5
90 minutes	23.0	5.75	6.00	2.7
SCOUR IV:				
Unacetylated	-	5.00	8.13	5.0
30 minutes	16.3	5.32	6.93	3.9
60 minutes	21.5	5.52	6.56	3.3
90 minutes	24.2	5.53	6.22	2.2

Table 9. Physical Data for Stoneville 2B Cotton Partially Acetylated After Different Methods of Scouring

Time of Acetylation	Acetyl (%)	Fiber Fineness (Micronaire)	Fiber Strength (Pressley)	Moisture Content (%)
SCOUR I:				
Unacetylated	-	3.50	7.72	4.8
30 minutes	16.2	3.73	6.66	2.6
60 minutes	20.4	4.15	6.23	2.8
90 minutes	23.6	4.18	5.96	1.8
SCOUR II:				
Unacetylated	-	3.53	8.00	4.8
30 minutes	16.3	3.63	6.82	2.3
60 minutes	20.2	4.00	6.28	2.7
90 minutes	25.0	4.03	5.92	1.8
SCOUR III:				
Unacetylated	-	3.50	7.99	5.1
30 minutes	16.8	3.80	6.63	3.4
60 minutes	22.5	4.05	6.05	3.6
90 minutes	23.8	4.15	5.79	2.7
SCOUR IV:				
Unacetylated	-	3.50	7.95	5.0
30 minutes	17.6	3.70	6.59	3.8
60 minutes	22.5	3.95	6.28	3.6
90 minutes	24.8	4.05	5.57	2.3

Table 10. Physical Data for Acala 1517 Cotton Partially Acetylated After Different Methods of Scouring

Time of Acetylation	Acetyl (%)	Fiber Fineness (Micronaire)	Fiber Strength (Pressley)	Moisture Content (%)
SCOUR I:				
Unacetylated	-	3.95	8.28	4.8
30 minutes	15.9	4.10	7.06	2.6
60 minutes	21.1	4.65	6.38	3.0
90 minutes	25.0	4.63	5.99	2.0
SCOUR II:				
Unacetylated	-	3.93	8.61	4.6
30 minutes	17.9	4.00	6.78	2.2
60 minutes	21.5	4.45	6.59	2.7
90 minutes	25.0	4.57	6.04	2.1
SCOUR III:				
Unacetylated	-	4.00	8.57	5.4
30 minutes	17.0	4.28	7.08	3.5
60 minutes	22.7	4.50	6.70	3.8
90 minutes	24.6	4.60	6.49	2.7
SCOUR IV:				
Unacetylated	-	3.95	8.19	5.0
30 minutes	18.0	4.18	6.71	4.0
60 minutes	23.3	4.47	6.77	3.5
90 minutes	26.0	4.40	6.59	2.3

Table 11. Physical Data for Lockett 140 Cotton Partially Acetylated After Different Methods of Scouring

Time of Acetylation	Acetyl (%)	Fiber Fineness (Micronaire)	Fiber Strength (Pressley)	Moisture Content (%)
SCOUR I:				
Unacetylated	-	5.65	7.13	4.7
30 minutes	15.6	5.67	6.49	2.6
60 minutes	19.8	6.18	6.05	2.8
90 minutes	24.4	6.20	5.29	2.0
SCOUR II:				
Unacetylated	-	5.57	7.24	4.4
30 minutes	15.8	5.65	6.51	2.1
60 minutes	20.4	6.20	6.38	2.6
90 minutes	25.6	6.07	5.64	1.7
SCOUR III:				
Unacetylated	-	5.70	7.14	4.8
30 minutes	15.4	5.97	6.17	3.2
60 minutes	20.8	6.10	5.72	3.4
90 minutes	23.8	6.10	5.56	2.6
SCOUR IV:				
Unacetylated	-	5.68	7.15	4.7
30 minutes	16.3	6.00	6.03	3.8
60 minutes	21.9	6.07	6.11	3.4
90 minutes	24.8	6.15	5.86	2.3

Table 12. Physical Data for Memphis Cotton Partially Acetylated After Moisture Conditioning at Different Relative Humidities

Time of Acetylation	Acetyl (%)	Fiber Fineness (Micronaire)	Fiber Strength (Pressley)	Moisture Content -(%)
15 PER CENT RELATIVE HUMIDITY:				
30 minutes	12.3	2.67	6.56	5.4
60 minutes	18.4	2.75	6.13	4.6
90 minutes	20.7	3.35	5.45	3.8
35 PER CENT RELATIVE HUMIDITY:				
30 minutes	12.2	2.67	6.53	5.2
60 minutes	17.6	2.68	6.14	4.5
90 minutes	20.9	3.22	5.81	4.1
50 PER CENT RELATIVE HUMIDITY:				
30 minutes	12.6	2.60	7.02	5.6
60 minutes	19.4	2.73	6.13	4.4
90 minutes	24.3	3.23	5.73	4.1
65 PER CENT RELATIVE HUMIDITY:				
30 minutes	14.0	2.67	6.59	4.6
60 minutes	19.5	2.80	6.14	4.1
90 minutes	23.5	3.02	5.93	4.7
85 PER CENT RELATIVE HUMIDITY:				
30 minutes	14.3	2.50	6.18	4.7
60 minutes	21.2	2.73	6.11	4.12
90 minutes	24.8	3.08	5.66	4.1

Table 13. Physical Data for Empire Bale 92 Cotton Partially Acetylated After Moisture Conditioning at Different Relative Humidities

Time of Acetylation	Acetyl (%)	Fiber Fineness (Micronaire)	Fiber Strength (Pressley)	Moisture Content (%)
15 PER CENT RELATIVE HUMIDITY:				
30 minutes	5.7	4.02	7.14	6.2
60 minutes	10.6	3.93	6.92	5.6
90 minutes	15.9	4.43	5.98	4.3
35 PER CENT RELATIVE HUMIDITY:				
30 minutes	5.39	3.93	7.44	5.6
60 minutes	9.67	3.97	6.87	5.7
90 minutes	15.3	4.25	6.63	4.2
50 PER CENT RELATIVE HUMIDITY:				
30 minutes	6.31	3.93	7.06	6.3
60 minutes	11.7	4.02	6.73	5.0
90 minutes	17.5	4.43	6.11	4.6
65 PER CENT RELATIVE HUMIDITY:				
30 minutes	8.0	3.90	6.83	5.1
60 minutes	13.5	4.02	6.47	4.4
90 minutes	18.8	4.30	5.99	4.7
85 PER CENT RELATIVE HUMIDITY:				
30 minutes	8.26	3.90	7.08	5.6
60 minutes	14.7	4.00	6.79	4.3
90 minutes	18.6	4.20	6.56	4.4

Table 14. Physical Data for Bob Shaw Cotton Partially Acetylated After Moisture Conditioning at Different Relative Humidities

Time of Acetylation	Acetyl (%)	Fiber Fineness (Micronaire)	Fiber Strength (Pressley)	Moisture Content (%)
15 PER CENT RELATIVE HUMIDITY:				
30 minutes	3.87	5.38	8.07	6.5
60 minutes	7.59	5.33	8.07	5.2
90 minutes	12.2	5.70	7.35	4.2
35 PER CENT RELATIVE HUMIDITY:				
30 minutes	3.57	5.33	8.47	5.4
60 minutes	6.89	5.33	8.28	5.1
90 minutes	12.6	5.53	7.86	4.7
50 PER CENT RELATIVE HUMIDITY:				
30 minutes	4.47	5.47	8.49	6.4
60 minutes	8.82	5.38	8.17	4.8
90 minutes	14.6	5.78	7.34	4.8
65 PER CENT RELATIVE HUMIDITY:				
30 minutes	5.95	5.38	8.22	5.3
60 minutes	10.6	5.43	7.80	5.0
90 minutes	15.6	5.52	7.26	4.9
85 PER CENT RELATIVE HUMIDITY:				
30 minutes	5.76	5.27	8.37	5.6
60 minutes	11.3	5.50	8.10	4.8
90 minutes	15.6	5.55	7.49	4.1

Table 15. Physical Data for Stoneville 2B Cotton Partially Acetylated After Moisture Conditioning at Different Relative Humidities

Time of Acetylation	Acetyl (%)	Fiber Fineness (Micronaire)	Fiber Strength (Pressley)	Moisture Content (%)
15 PER CENT RELATIVE HUMIDITY:				
30 minutes	6.59	3.92	8.19	5.9
60 minutes	11.7	3.80	7.51	5.2
90 minutes	16.8	4.28	6.91	3.9
35 PER CENT RELATIVE HUMIDITY:				
30 minutes	6.54	3.80	8.25	5.7
60 minutes	10.7	3.73	8.07	5.3
90 minutes	17.1	4.10	7.35	4.5
50 PER CENT RELATIVE HUMIDITY:				
30 minutes	7.64	3.75	8.21	6.1
60 minutes	13.0	3.82	7.91	5.2
90 minutes	19.2	4.22	6.90	4.3
65 PER CENT RELATIVE HUMIDITY:				
30 minutes	9.37	3.80	7.80	5.0
60 minutes	15.1	3.85	7.17	4.5
90 minutes	19.2	4.10	6.34	4.4
85 PER CENT RELATIVE HUMIDITY:				
30 minutes	9.81	3.68	8.14	5.4
60 minutes	16.3	3.80	7.37	4.6
90 minutes	20.4	4.08	6.47	4.1

Table 16. Physical Data for Acala 1517 Cotton Partially Acetylated After Moisture Conditioning at Different Relative Humidities

Time of Acetylation	Acetyl (%)	Fiber Fineness (Micronaire)	Fiber Strength (Pressley)	Moisture Content (%)
15 PER CENT RELATIVE HUMIDITY:				
30 minutes	6.51	4.30	8.68	6.4
60 minutes	11.3	4.33	8.21	5.4
90 minutes	17.0	4.95	7.08	4.3
35 PER CENT RELATIVE HUMIDITY:				
30 minutes	6.90	4.25	8.34	5.9
60 minutes	11.8	4.27	8.36	5.3
90 minutes	17.3	4.67	7.11	4.4
50 PER CENT RELATIVE HUMIDITY:				
30 minutes	8.25	4.25	8.20	6.2
60 minutes	14.0	4.52	8.02	5.4
90 minutes	19.7	4.90	7.01	4.7
65 PER CENT RELATIVE HUMIDITY:				
30 minutes	7.95	4.20	8.70	5.2
60 minutes	14.0	4.33	7.76	4.9
90 minutes	20.3	4.55	6.74	4.8
85 PER CENT RELATIVE HUMIDITY:				
30 minutes	11.4	4.20	8.48	5.7
60 minutes	19.1	4.43	7.36	4.4
90 minutes	22.9	4.88	6.94	4.0

Table 17. Physical Data for Lockett 140 Cotton Partially Acetylated After Moisture Conditioning at Different Relative Humidities

Time of Acetylation	Acetyl (%)	Fiber Fineness (Micronaire)	Fiber Strength (Pressley)	Moisture Content (%)
15 PER CENT RELATIVE HUMIDITY:				
30 minutes	3.40	6.00	7.40	6.4
60 minutes	6.84	6.00	7.12	5.8
90 minutes	11.7	6.38	6.79	4.7
35 PER CENT RELATIVE HUMIDITY:				
30 minutes	3.51	6.05	6.84	5.8
60 minutes	6.87	5.98	7.18	6.0
90 minutes	11.4	6.30	7.14	4.8
50 PER CENT RELATIVE HUMIDITY:				
30 minutes	3.90	6.00	7.84	6.3
60 minutes	8.51	5.95	7.16	5.5
90 minutes	13.0	6.17	6.58	5.2
65 PER CENT RELATIVE HUMIDITY:				
30 minutes	6.32	5.95	7.05	5.4
60 minutes	10.8	5.95	6.65	4.9
90 minutes	13.5	6.22	6.51	5.2
85 PER CENT RELATIVE HUMIDITY:				
30 minutes	6.84	5.93	7.06	5.9
60 minutes	12.2	6.07	6.86	4.9
90 minutes	16.2	6.13	6.55	4.5

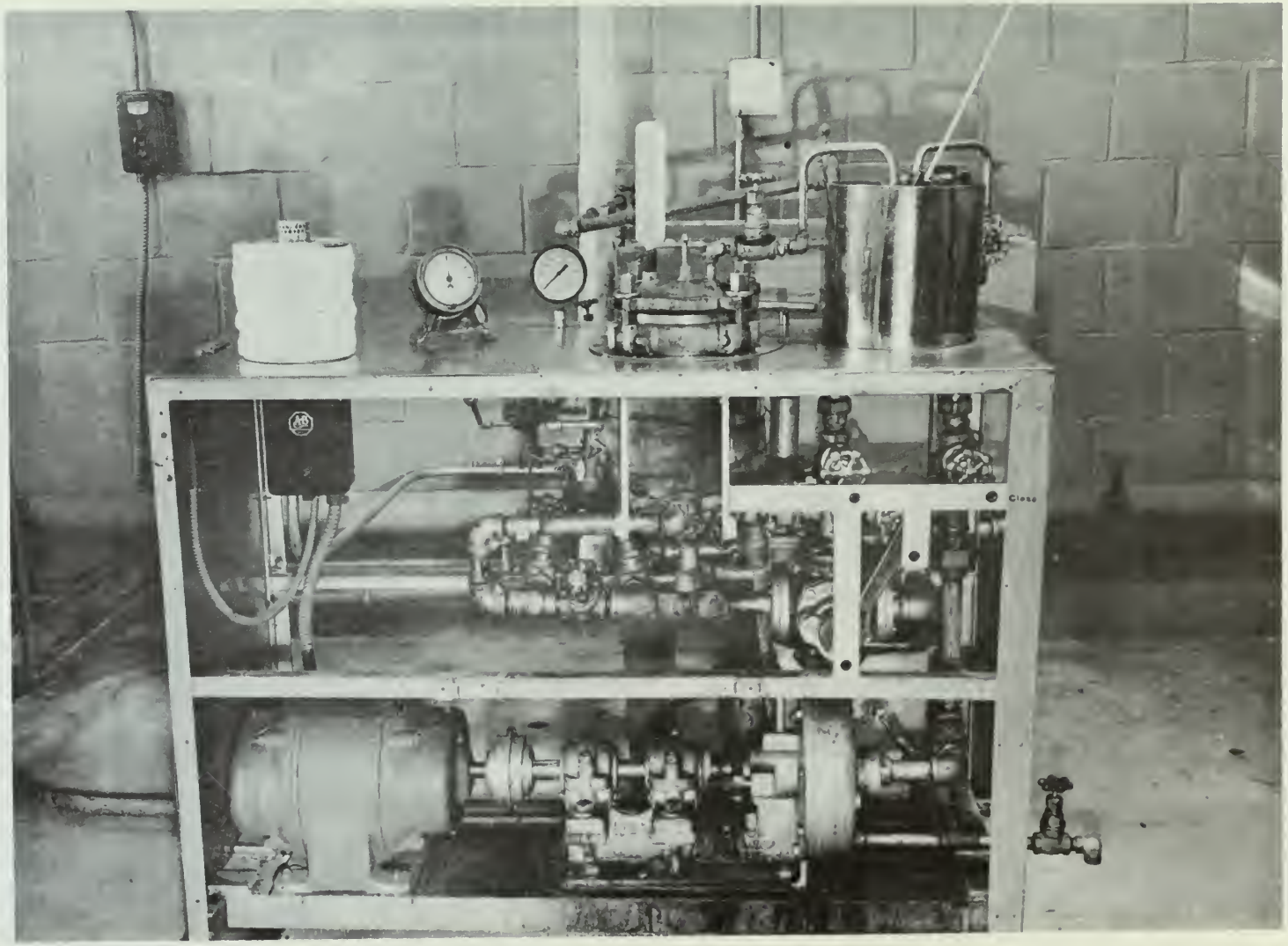

Figure 24. Front View of Acetylation Machine.

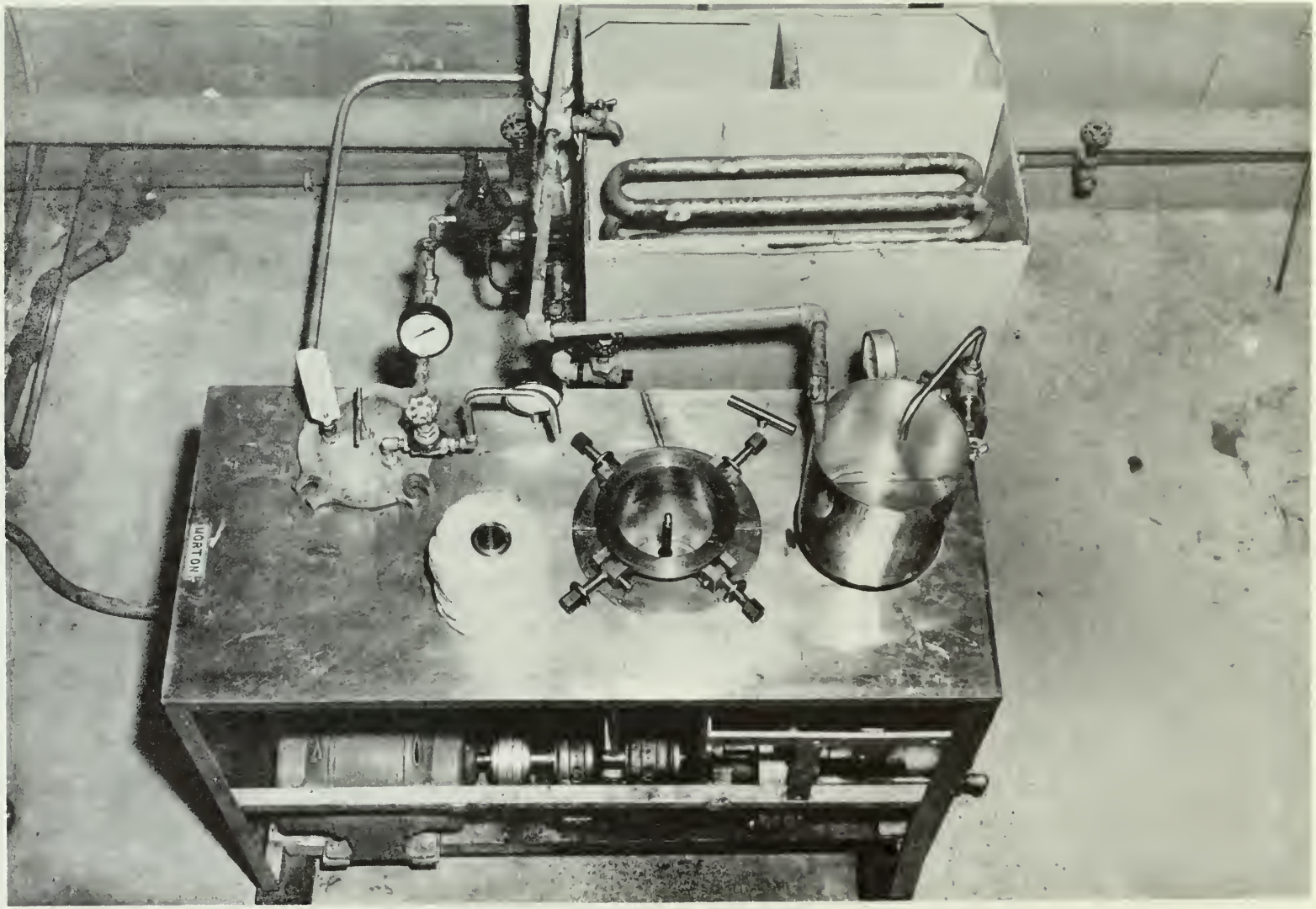

Figure 25. Top View of Acetylation Machine.